Vol. XXIII November, 1946 No. 6

CEREAL CHEMISTRY

Published bimonthly by the American Association of Cereal Chemists
at Prince and Lemon Sts., Lancaster, Pa.

CONTENTS

Manuscripts for publication should be sent to the Editor in Chief. Advertising rates may be secured from and subscriptions placed with the Managing Editor, University Farm, St. Paul 1, Minnesota. Subscription rates, $6 per year. Foreign postage, 50 cents extra. Single copies, $1.25; foreign, $1.35.

Entered as second-class matter March 3, 1932, at the post office at Lancaster, Pa., under the act of August 24, 1912.

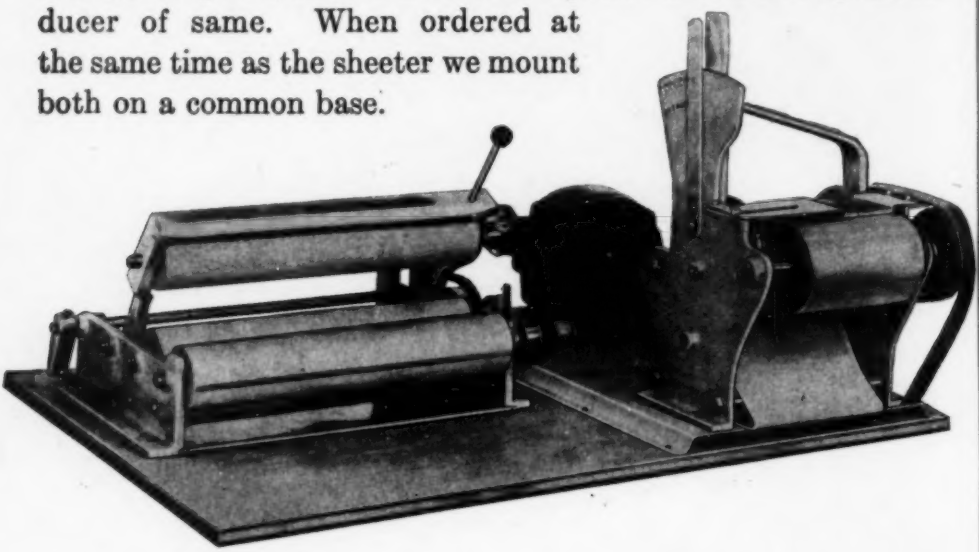
Acceptance for mailing at special rate of postage provided for in Section 1103, Act of October 3, 1917, authorized February 16, 1924.

SHEETING ROLLS

for the TEST BAKING LABORATORY

We manufacture two sizes of sheeting rolls; the 3″ width for Pup Loaves and the 6″ width or 1 lb. size for all loaves from Pup to Standard loaves, which model is shown below mounted with three-roll-type moulder.

Our Three-Roll-Type Moulder can be attached to either size sheeting roll, and is driven from power take-off of speed reducer of same. When ordered at the same time as the sheeter we mount both on a common base.



UNIVERSAL "THREE-ROLL-TYPE" MOULDER

PRICE $80.00

Test Bake Moulding can be done mechanically with the operator confident that his doughs are:

1. Moulded and sealed in an effective and GENTLE manner,
2. Ends of doughs are not abused and
3. Entire operation on dough is visible at all times.
4. Variation between operators is reduced.

1 LB. SIZE POWER SHEETING ROLL

as shown above, including G.E. 110 V., A.C. motor, speed reducer and foot switch.

PRICE $160.00

PUP SIZE POWER SHEETING ROLL

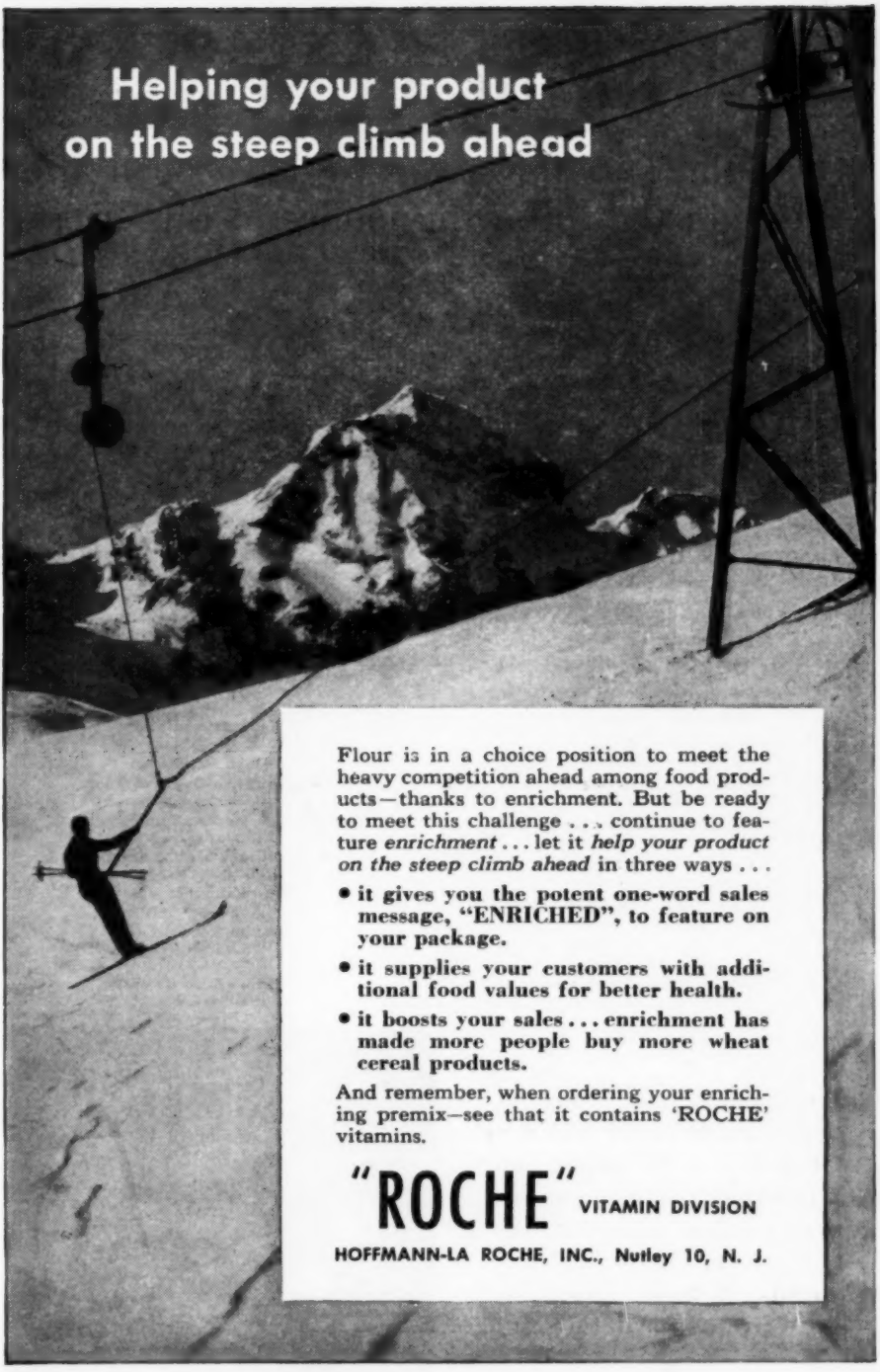
Same as above but 3″ width

PRICE $125.00

CEREAL CHEMISTS' EQUIPMENT MANUFACTURER

NATIONAL MFG. COMPANY

LINCOLN, NEBRASKA



Flour is in a choice position to meet the heavy competition ahead among food products—thanks to enrichment. But be ready to meet this challenge . . . continue to feature *enrichment* . . . let it *help your product on the steep climb ahead* in three ways . . .

- **it gives you the potent one-word sales message, "ENRICHED", to feature on your package.**
- **it supplies your customers with additional food values for better health.**
- **it boosts your sales . . . enrichment has made more people buy more wheat cereal products.**

And remember, when ordering your enriching premix—see that it contains 'ROCHE' vitamins.

"ROCHE" VITAMIN DIVISION

HOFFMANN-LA ROCHE, INC., Nutley 10, N. J.

We are justifiably proud of the evergrowing list of

FARINOGRAPH *users:*

Over the past few years we have been able to add the following names to this list:

American Flours, Inc.
Newton, Kan.

American Institute of Baking
Chicago, Ill.

Anheuser-Busch, Inc.
St. Louis, Mo.

H. C. Cole Milling Company
Chester, Ill.

Colorado Milg. & Elev. Co.
Denver, Colo.

Consolidated Flour Mills
Wichita, Kan.

Doty Technical Laboratories
Kansas City, Mo.

Valier & Spies Milling Co.
St. Louis, Mo.

Kansas Flour Mills Company
North Kansas City, Mo.

Higginsville Flour Mill
Higginsville, Mo.

Hunter Milling Company
Wellington, Kan.

Inland Mills, Inc.
Des Moines, Iowa

International Milling Co.
Detroit, Mich.

William Kelly Milling Co.
Hutchinson, Kan.

Kelly-Erickson Company
Omaha, Neb.

Langendorf United Bakeries
San Francisco, Cal.

Joe Lowe Corporation
New York City

Joe Lowe Corporation
Chicago, Ill.

Maney Milling Company
Omaha, Neb.

Mennel Milling Company
Toledo, Ohio

Midland Flour Milling Co.
Kansas City, Mo.

Montana Flour Mills Co.
Great Falls, Mont.

National Biscuit Company
Cheney, Wash.

National Tea Company
Chicago, Ill.

New Era Milling Company
Arkansas City, Kan.

Omar, Inc.
Omaha, Neb.

C. J. Patterson Company
Kansas City, Mo.

Pillsbury Mills Company (2nd Inst.)
Minneapolis, Minn.

Quality Bakers of America
New York City

Rodney Milling Company (2nd Inst.)
Kansas City, Mo.

Russell-Miller Milling Company
Alton, Ill.

Russell-Miller Milling Company (2nd Inst.)
Minneapolis, Minn.

F. W. Stock & Sons, Inc.
Hillsdale, Mich.

Vanderbilt Foods Corporation
East Norwalk, Conn.

Wallace & Tiernan Company, Inc.
Newark, N. J.

Walnut Creek Milling Company
Great Bend, Kansas

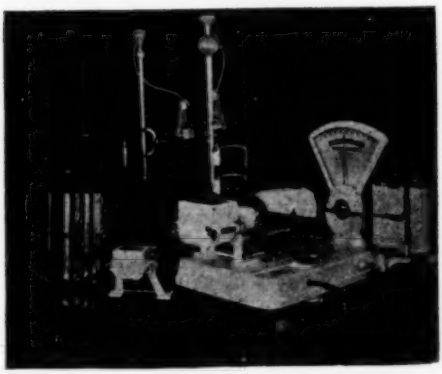
Ward Baking Company
New York City

Washburn Crosby Div.
General Mills, Inc.
Kansas City, Mo.

Williams Bros. Company
Kent, Ohio

●

You, too, can have one of the latest model FARINOGRAPHS. We have an attractive rental proposition so that you may try the instrument thoroughly before purchase. Write us for details.



●

BRABENDER CORPORATION Rochelle Park, N. J.

Imagine You

WEARING AN ASPHALT RAINCOAT!

• Probably you'd look terrible. But one thing is sure—you'd stay *dry!* Asphalt keeps out moisture. That's why we use asphalt to help keep Diamond Crystal Salt dry, and prevent caking.

Salt usually cakes when exposed to excessive humidity, because moisture condenses on salt particles, forming a thin layer of brine. Then, in dry weather, the brine evaporates and the crystals knit together.

Our research laboratory has found a number of ways to help prevent salt caking. Removal of moisture-attracting impurities, such as calcium chloride, helps. So does complete removal of fines by careful screening. Most important is to provide salt with a moisture-vapor-resistant package.

Take our Flour Salt bag, for example. It is composed of three 50-lb. and two 25-lb. sheets of kraft, laminated together with 40 lbs. of asphalt per ream. That's a lot of asphalt, and it costs us more money—but our moisture-vapor transmission tests show that this bag is well worth the extra cost. The bag is even sewn with *waxed thread* to seal the holes made by the sewing-machine needle. That's real *protection*—one reason we have been able to eliminate caking as a major problem!

WANT FREE INFORMATION ON SALT? WRITE US!

If *you* have a salt problem, write to our Director of Technical Service. He will be happy to help! Diamond Crystal Salt, Dept. M-13, St. Clair, Michigan.

DIAMOND CRYSTAL SALT

ALBERGER PROCESS

Why your Dow distributor is your best fumigation source

Food processors and vendors everywhere depend more and more on the *reliable* fumigant distributor.

That's because fumigation is *more* than a business. It's a technique—a way of achieving fullest protection against marauding insects and rodents which would otherwise become an unbearable problem to the entire food industry—and to the nation.

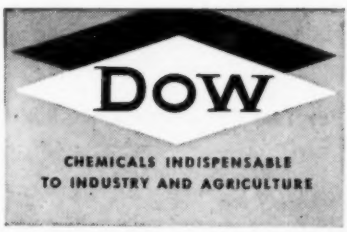
To distributors of Dow fumigants—responsible men who *know* fumigation—have come many requests for fumigation services. The reason is clear: Distributors, operators and engineers—with their own reputations at stake on every job—have found in DOW METHYL BROMIDE, DOW CHLOROPICRIN and other Dow fumigants dependable quality which matches their own high standards of service.

Outstanding distributors and operators know that they can safely recommend Dow fumigants to their best customers because each product is designed to do the job and do it right.

No wonder fumigation customers say: "The way to get *good* fumigation is to look for the reliable supplier who offers *Dow* fumigants!"

WHEREVER RESULTS COUNT—
DEPEND ON THE FUMIGANT DEALER
WHO KNOWS!

THE DOW CHEMICAL COMPANY • MIDLAND, MICH.
New York • Boston • Philadelphia • Washington • Cleveland • Detroit
Chicago • St. Louis • Houston • San Francisco • Los Angeles • Seattle



STRENGTH

Robert W. Brooks, Manager of Product Service, Fleischmann Division of Standard Brands Incorporated, who has been with the organization for 25 years, has this to say about strength in yeast.

High quality baked goods depend on yeast strength.

Every baker knows that strength in yeast means its power to initiate and sustain the entire process of dough fermentation until this action is terminated by oven heat.

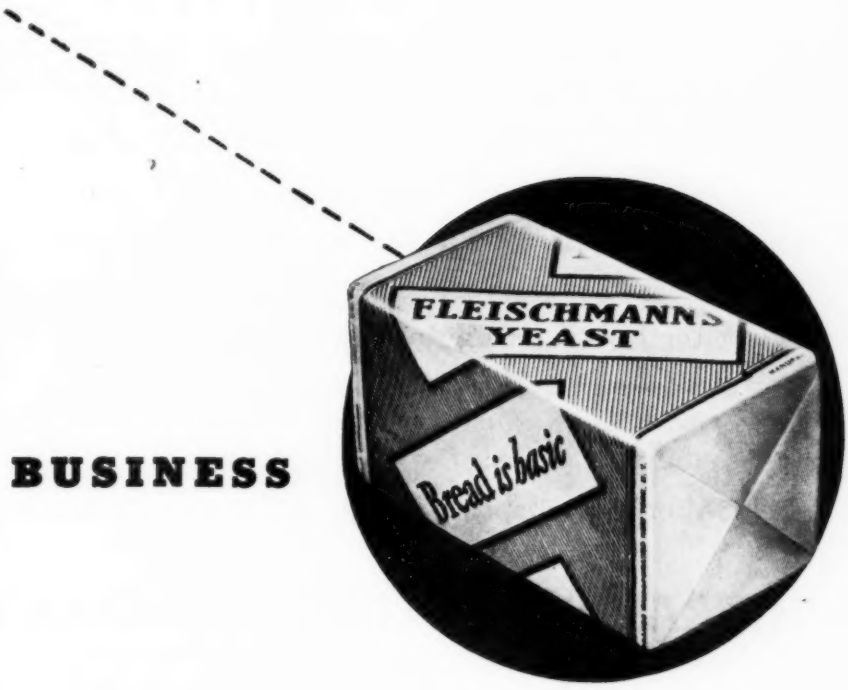
Experience plus knowledge and scientific control assure the strength of Fleischmann's Yeast.

Our Plant Managers are experienced men. For our plant staffs we constantly add carefully selected young men with thorough scientific background.

They are the guardians of the Strength bakers depend upon in Fleischmann's Yeast.

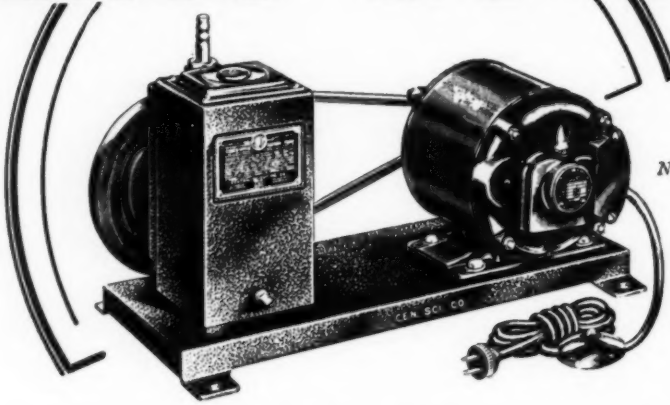
FERMENTATION IS OUR

Buy Your Extra Bond Now—Buy Through Pay-roll Savings



BUSINESS

CENCO HYVAC



No. 91105A

Years of Service

are built into Cenco Hyvac Pumps. They give a consistently high vacuum of 0.3 micron or better. Cenco Hyvac Pumps are renowned for faithful and long service. They have proven their ability to perform continuously under heavy production conditions. These sturdy qualities are consistently uniform in each and every pump. Laboratory men prefer the Hyvac for its small size, fast action, ready portability and its freedom from adjustments or repairs.

91105A for 115 volts, 60 cycles A. C. $75.00

CENTRAL SCIENTIFIC COMPANY

SCIENTIFIC INSTRUMENTS CENCO LABORATORY APPARATUS

NEW YORK TORONTO CHICAGO BOSTON SAN FRANCISCO

CEREAL CHEMISTRY

VOL. XXIII NOVEMBER, 1946 No. 6

EFFECT OF PARBOILING ON THIAMINE IN RICE [1]

M. C. KIK

Agricultural Chemistry Department, University of Arkansas, Fayetteville, Arkansas

(Received for publication June 25, 1946)

The process of parboiling consists essentially of soaking rough rice in cold, warm, or hot water followed by draining the water, steaming the rice, and drying. The hypothesis is that during such treatment the water-soluble vitamins diffuse from the outer layers of the kernel to the inner layers and more of these water-soluble nutrients are retained in the kernel.

The parboiling, as practiced in the Orient, is by no means standardized. In Malaya (Jack, 1923), the paddy is merely heated by steam under pressure, quickly dried, and milled. In 1935 Jones and Taylor concluded from their experimental studies that only 12 hours of soaking at 60°–70°C, followed by steaming, are required to give a good product. A review of these methods is given by Kik (1945).

Modern commercial practices of parboiling find application in the process of rice conversion, in the Malek parboiling process (Kik and Williams, 1945), and in the Sacramento Mill in California where rough rice is soaked and exposed to steam under pressure.

Data have been published on the effect of parboiling and milling on the thiamine content of rice (Aykroyd, 1932) and on milling quality (Jones and Taylor, 1935; Subrahmanyan, Sreenivasan, and Das Gupta, 1938). No extensive study has been made of the thiamine content of rice which has been parboiled according to simple controlled methods in which modern analytical procedures for the determination of the thiamine content have been employed.

This investigation deals with the effect of simple controlled methods of parboiling of rough rice on the thiamine content of the milled form after approximately the same percent of the bran (10%) has been

[1] Research paper No. 813, Journal Series, University of Arkansas. Published with the permission of the Director of the Arkansas Agricultural Experiment Station. Aided by grants from the Williams-Waterman Fund of the Research Corporation and of Converted Rice. A preliminary report has been presented at the meeting of the American Institute of Nutrition, which was held in Atlantic City, New Jersey, on March 11–15, 1946.

removed. Shelling tests were made on all these samples, which showed the breakage during shelling. Data are also presented on the effect of soaking for different lengths of time on the thiamine content of milled rough and brown rice and of losses of this vitamin in the soaking water.

Materials and Methods

A total of 88 samples (weight 60 g) of the same lot of previously cleaned rough rice, medium grain (Zenith variety), were soaked in 150 ml of distilled water under varying conditions of duration and temperature and with variations in amounts of steam pressure. These samples of rough rice were parboiled by boiling; boiling and steaming; steaming; soaking and steaming; soaking, boiling, and steaming; according to rice conversion with vacuum; and according to rice conversion without vacuum. The details of these treatments are given in Tables I and II. After the treatment, the water was drained off,

TABLE I

EFFECT OF DIFFERENT METHODS OF PARBOILING ROUGH RICE (ZENITH VARIETY) ON THIAMINE [1] IN MILLED RICE AND ON BREAKAGE [2] DURING SHELLING
(Milled to approximately 10% bran removal)

Sample No.	Treatment	Thiamine	Appearance of the milled product	Breakage
		μg/g		%
1	No treatment	0.66	Opaque	3.6
2	No treatment	0.40	Opaque	4.1
3	No treatment	0.79	Opaque	3.6
4	No treatment	0.56	Opaque	3.9
	Average	0.60		3.8
	a. *Boiling:*			
5	For 5 minutes	2.39	Opaque	45.2
6	For 10 minutes	2.31	Opaque	40.6
7	For 15 minutes	2.24	Opaque	27.6
	Average	2.31		37.8
	b. *Boiling and Steaming:*			
8	Boiled 5 min., steamed 10 min. at 5 lbs. pres.	2.40	Opaque	6.0
9	Boiled 5 min., steamed 10 min. at 10 lbs. pres.	2.14	Opaque	6.0
10	Boiled 5 min., steamed 10 min. at 15 lbs. pres.	2.04	Opaque	10.2
11	Boiled 10 min., steamed 10 min. at 5 lbs. pres.	2.36	Opaque	12.0
12	Boiled 10 min., steamed 10 min. at 10 lbs. pres.	2.25	Opaque	13.8
13	Boiled 10 min., steamed 10 min. at 15 lbs. pres.	2.00	Partly translucent	2.4
14	Boiled 10 min., steamed 15 min. at 10 lbs. pres.	2.34	Opaque	2.6
15	Boiled 15 min., steamed 15 min. at 10 lbs. pres.	2.61	Opaque	4.4
16	Boiled 20 min., steamed 15 min. at 10 lbs. pres.	2.13	Translucent	4.0
	Average	2.25		6.8

TABLE I—(*Continued*)

Sample No.	Treatment	Thiamine	Appearance of the milled product	Breakage
		μg/g		%
	c. *Steaming:*			
17	10 minutes at 5 lbs. pres.	2.36	Opaque	27.1
18	20 minutes at 5 lbs. pres.	2.43	Opaque	13.2
19	30 minutes at 5 lbs. pres.	2.44	Opaque	9.7
20	5 minutes at 10 lbs. pres.	1.96	Opaque	11.3
21	10 minutes at 10 lbs. pres.	2.05	Opaque	10.1
22	15 minutes at 10 lbs. pres.	2.27	Opaque	3.5
23	20 minutes at 10 lbs. pres.	2.40	Opaque	7.6
24	5 minutes at 15 lbs. pres.	2.40	Opaque	52.7
25	10 minutes at 15 lbs. pres.	2.33	Opaque	29.2
26	20 minutes at 15 lbs. pres.	2.54	Opaque	8.4
27	5 minutes at 20 lbs. pres.	2.46	Opaque	52.6
28	10 minutes at 20 lbs. pres.	2.40	Opaque	8.5
29	15 minutes at 20 lbs. pres.	2.19	Opaque	4.8
	Average	2.51		
	d. *Soaking, Boiling, and Steaming:*			
83	2 hours soaking at 27°C, boiled 5 min., steamed 10 min. at 15 lbs. pres.	2.34	Opaque	15.1
84	2 hours soaking at 27°C, boiled 10 min., steamed 10 min. at 10 lbs. pres.	2.32	Opaque	46.7
85	4 hours soaking at 27°C, boiled 5 min., steamed 10 min. at 15 lbs. pres.	2.01	Translucent	2.0
86	4 hours soaking at 27°C, boiled 10 min., steamed 10 min. at 15 lbs. pres.	2.30	Translucent	2.5
	e. According to the principle of rice conversion:			
87	30 min. vacuum, pressure 60 lbs./inch at 70°C for 2½ hours.	2.48	Translucent	3.5
88	No vacuum, pressure 60 lbs. at 70°C for 2½ hours.	2.35	Translucent	3.5

[1] Expressed on a basis of 9% moisture. [2] Determined with Smith's Shelling Device.

the rough rice was dried overnight at room temperature with the aid of a fan, shelled, milled to approximately 10% bran removal, and assayed for thiamine. Four nontreated samples were used as controls.

In the final evaluation of the efficiency of the parboiling process, the appearance of the rice kernel after milling and the percentage of breakage during shelling, as well as the retention of thiamine in the milled product, have to be taken into consideration, since these properties are of paramount economic importance in the industrial application of these results.

The shelling and milling were done with laboratory-scale equipment. The seed huller [2] was used for the removal of hulls from the

[2] Purchased from the Seedburo Equipment Company, 223 West Jackson Boulevard, Chicago 6, Illinois.

rough rice, and the small hand barley pearler [2] was employed for the milling of brown rice to 10% bran removal.

The rice was steamed under pressure in a small pressure cooker. The pressure ranged from 5 to 20 pounds and the temperature from 104°C to 127°C. The laboratory processing of rice according to the

TABLE II

EFFECT OF SOAKING AND STEAMING OF ROUGH RICE (ZENITH VARIETY) ON THIAMINE IN MILLED RICE AND ON BREAKAGE DURING SHELLING
(Milled to approximately 10% bran removal)

Sample No.	Treatment	Thiamine	Appearance of the milled product	Breakage
		μg/g		%
	a. Soaking for 15 hours at room temperature (27°C) followed by:			
30	5 min. steaming at 15 lbs. pres.	2.48	Partly translucent	3.5
31	10 min. steaming at 15 lbs. pres.	2.14	Translucent	6.1
32	15 min. steaming at 15 lbs. pres.	2.29	Translucent	5.2
33	20 min. steaming at 15 lbs. pres.	2.13	Translucent	9.1
34	25 min. steaming at 15 lbs. pres.	2.71	Translucent	6.1
35	30 min. steaming at 15 lbs. pres.	2.22	Translucent	8.8
36	35 min. steaming at 15 lbs. pres.	2.32	Translucent	6.7
37	40 min. steaming at 15 lbs. pres.	2.00	Translucent	3.2
	Average	2.28		6.1
	b. Soaking for 45 hours at room temperature (27°C) followed by:			
38	10 min. steaming at 15 lbs. pres.	2.25	Translucent	0.9
39	15 min. steaming at 15 lbs. pres.	2.26	Translucent	1.7
40	20 min. steaming at 15 lbs. pres.	2.25	Translucent	2.8
41	25 min. steaming at 15 lbs. pres.	1.97	Translucent	4.7
42	30 min. steaming at 15 lbs. pres.	1.73	Translucent	5.7
43	35 min. steaming at 15 lbs. pres.	1.99	Translucent	3.8
	Average	2.07		3.2
	c. Soaking for indicated length of time at 35°C, followed by 10 min. steaming at 15 lbs. pres.			
44	2 hours	1.93	Opaque	3.1
45	4 hours	2.21	Slightly opaque	3.0
46	6 hours	2.09	Translucent	3.3
47	8 hours	2.24	Translucent	6.5
48	10 hours	2.80	Translucent	2.9
49	12 hours	2.40	Translucent	3.8
50	16 hours	2.30	Translucent	2.1
51	20 hours	2.08	Translucent	2.3
52	24 hours	2.57	Translucent	1.0
53	36 hours	2.57	Translucent	2.7
	Average	2.29		3.1
	d. Soaking for 6 hours at 40°C followed by:			
54	15 min. steaming at 15 lbs. pressure (Malekized process)	2.25	Translucent	0.9

TABLE II—(*Continued*)

Sample No.	Treatment	Thiamine	Appearance of the milled product	Breakage
		μg/g		%
	e. Soaking for indicated length of time at 60°C, followed by 10 min. steaming at 15 lbs. pres.			
55	2 hours	1.91	Translucent	2.9
56	4 hours	2.27	Fine translucent	1.9
57	6 hours	2.16	Fine translucent	2.3
58	8 hours	2.16	Fine translucent	3.1
59	10 hours	2.07	Fine translucent	5.9
60	12 hours	2.28	Fine translucent	2.6
61	16 hours	2.31	Translucent	2.2
62	20 hours	2.26	Translucent	3.8
63	24 hours	2.27	Fine translucent	4.3
64	36 hours	1.93	Fine translucent	3.8
	Average	2.16	•	3.3
	f. Soaking for 2 hours at 70°C, followed by:			
65	5 min. steaming at 15 lbs. pres.	2.91	Opaque	6.1
66	10 min. steaming at 15 lbs. pres.	2.66	Slightly translucent	6.3
67	15 min. steaming at 15 lbs. pres.	2.72	Fair translucent	6.6
68	20 min. steaming at 15 lbs. pres.	2.64	Opaque	5.7
	Average	2.73		6.2
	g. Soaking for indicated time at 70°C, followed by: 10 min. steaming at 15 lbs. pres.			
69	1 hour	2.38	Opaque	2.3
70	2 hours	2.57	Slightly translucent	2.0
71	3 hours	2.46	Translucent	6.7
72	4 hours	2.88	Translucent	3.9
73	5 hours	2.48	Fine translucent	3.3
74	6 hours	2.34	Translucent	3.0
	Average	2.52		3.5
	h. Soaking for indicated time at 80°C, followed by: 10 min. steaming at 15 lbs. pres.			
75	½ hour	2.44	Slightly opaque	3.5
76	1 hour	2.48	Opaque	5.4
77	2 hours	2.58	Translucent	13.0
78	3 hours	2.29	Translucent	14.6
	Average	2.45		9.1
	i. Soaking for indicated time at 90°C, followed by: 10 min. steaming at 15 lbs. pres.			
79	10 minutes	2.52	Slightly translucent	3.6
80	20 minutes	2.51	Slightly translucent	10.3
81	30 minutes	2.10	Translucent	6.8
82	40 minutes	2.35	Translucent	4.9
	Average	2.37		6.4

principles of rice conversion in which 60 pounds pressure was applied has been described in a previous publication (Kik, 1945).

In soaking experiments, eleven 100-g samples of previously cleaned rough and brown rice (Zenith, medium grain variety) were soaked from 0 to 36 hours in 150 ml of distilled water. The rice was dried, shelled, milled to approximately 10% bran removal, and tested for thiamine by the thiochrome method, with the adaptation of the Hennessy and Cerecedo procedure (1939). The thiamine results are expressed on a basis of 9% moisture. The results of these tests are given in Table III.

TABLE III

EFFECT OF TIME OF SOAKING OF BROWN AND ROUGH RICE ON THE THIAMINE CONTENT OF MILLED RICE

Sample No.	Time of soaking	Average thiamine content of milled rice	Loss of thiamine in steep water
	hours	μg/g	%
SOAKING OF BROWN RICE			
1	0	0.44	0.00
2	8	1.03	10.90
3	12	1.08	14.24
4	24	0.91	15.80
5	36	0.79	16.00
SOAKING OF ROUGH RICE			
6	4	1.35	1.71
7	8	1.03	1.30
8	12	1.12	4.04
9	16	1.50	2.01
10	24	1.37	2.63
11	36	1.58	1.86

Results

Parboiling Tests. The data relating to parboiling tests are presented in Tables I and II. The nontreated controls, numbers 1, 2, 3, and 4, had an average content of thiamine of 0.60 μg/g and the average breakage on shelling was 3.8%.

BOILING: Three samples of rough rice (numbers 5, 6, 7) were exposed to boiling water for 5, 10, and 15 minutes respectively and showed a considerable retention of thiamine (average content 2.31 μg/g) which was not affected by the time of exposure to boiling water. However, excessive breakage (average 37.8%) occurred during shelling, which makes the treatment of boiling alone rather unsatisfactory. Exposure to boiling water for 15 minutes (sample 7) retarded the

breakage to 27.6%. This showed that prolonged boiling helped reduce breakage.

BOILING AND STEAMING: A total of nine samples, numbers 8 to 16 inclusive, was used (see *b* Table I). Samples 8, 9, and 10 were boiled for 5 minutes followed by steaming for 10 minutes at pressures ranging from 5 to 15 pounds (109°C–121°C). The milled samples were opaque in appearance like ordinary milled rice, whereas parboiled rice is translucent to amber, and generally of a straw-yellow color. The translucence is due to the complete gelatinization of the starch which takes place at temperatures varying from 60° to 75°C (Jones and Taylor, 1935). Some of the kernels in these samples showed a white spot in the center of the grain due to partial gelatinization of the starch.

The additional steaming reduced the breakage from an average of 37.8% in boiling tests to 6.8% in boiling and steaming tests. This indicates that steaming decreases the breakage. The lowest breakage (2.4%) was obtained after the steam pressure was increased from 5 and 10 pounds (samples 11 and 12) to 15 pounds (sample 13). Fairly well parboiled rice was obtained in sample 16 which was boiled for 20 minutes and steamed afterwards for 15 minutes at 5 pounds pressure. The average thiamine content in these samples was 2.25 μg/g, which showed that additional steaming did not have any beneficial effect. Prolonged boiling did not have any effect on the thiamine content of the milled product.

STEAMING: The rough rice was previously soaked for 10 minutes in water of room temperature, as dry starch cannot be gelatinized and the kernels are brittle after steaming. A total of 13 samples was steamed at pressures ranging from 5 to 20 pounds for different lengths of time. The average thiamine content of the milled samples was 2.51 μg/g (see Table I). The breakage was appreciably reduced by proper steaming. A breakage of 27.1% was reduced to 9.7% when 10 minutes' steaming at 5 pounds pressure (sample 17) was increased to 30 minutes (sample 19). Similar comparisons can be made with samples 24 and 26, samples 27 and 29. All kernels were still opaque after the treatment, which indicates that steaming after only a brief soaking in water of room temperature does not give satisfactory results.

SOAKING AND STEAMING: *a.* Eight samples, numbers 30 to 37, were *soaked for 15 hours at room temperature (27°C).* The samples were steamed afterwards at 15 pounds pressure for a duration ranging from 5 minutes for sample 30 to 40 minutes for sample 37. The results are presented in Table II. The average thiamine content was 2.28 μg/g; the average breakage was 6.1%. All kernels were translucent except those of number 30. The best results were obtained in number 34 which was steamed 25 minutes at 15 pounds pressure.

b. Another six samples, numbers 38 to 43, were *soaked at room temperature* (27°C) for 45 hours (See *b* Table II). The samples were steamed at 15 pounds pressure for from 10 minutes for sample 38 to 35 minutes for sample 43. Nice translucent kernels were obtained which showed an average breakage of 3.2%. Number 38, which showed the best results, had a thiamine content of 2.25 μg/g and a breakage of 0.9%. This sample was steamed for 10 minutes at 15 pounds pressure and the kernels were fully translucent in the milled state. A small decrease in thiamine content and a small increase in breakage were found in those samples (41, 42, and 43) which were steamed more than 20 minutes. In separate tests it was found that thiamine was partly destroyed when exposed for over 20 minutes at 15 pounds pressure.

c. The next series of samples was *soaked 2, 4, 6, 8, 10, 12, 16, 20, 24, and 36 hours in water of 35°C* followed by steaming for 10 minutes at 15 pounds pressure. In all 10 cases (numbers 44 to 53) except number 44, translucent kernels were obtained which showed an average breakage on shelling of 3.1% and an average thiamine content of 2.28 μg/g. Good results were found in all samples after a minimum of 6 hours of soaking. Number 52 showed a thiamine content of 2.57 μg/g, a breakage of 1%, and translucent kernels after milling. This sample was soaked for 24 hours before the steam treatment was applied.

d. One sample, number 54, was *soaked for 6 hours at 40°C* followed by 15 minutes steaming at 15 pounds pressure. This method is applied in the Malekized process which is used on a commercial scale. The sample was milled to the same degree of bran removal as all other samples and showed a thiamine value of 2.25 μg/g. The kernels were translucent and an exceedingly low breakage of 0.9% was obtained during shelling.

e. The next series consisted of *10 samples which were soaked at 60°C for 2, 4, 6, 8, 10, 12, 16, 20, 24, and 36 hours respectively.* In all cases translucent kernels were found in the milled product. The average thiamine content in this series was 2.16 μg/g and the average breakage was 3.3%. However, best results were obtained from samples soaked a minimum of 4 hours.

f. Soaking for 2 hours at 70°C followed by steaming at 15 pounds pressure took place in four samples, numbers 65 to 68, which were steamed for 5, 10, 15, and 20 minutes. These kernels were not quite translucent after milling and the average breakage was 6.2%, which was higher than the 3.8% found for the nontreated controls. However, the average thiamine content was 2.73 μg/g, which is higher than has been found in milled samples exposed to other treatments.

g. Six samples were *soaked at 70°C followed by 10 minutes at 15 pounds pressure* for 1, 2, 3, and 6 hours respectively. The results were good after the rice had been soaked for more than 2 hours. This can be seen from Table II (samples 69, 70, 71, 72, 73, and 74). The first two samples showed kernels that were not entirely translucent. The remaining four samples were well parboiled. The average thiamine content of this group was 2.52 μg/g, with an average breakage of 3.5%. It would appear that soaking for 4 hours at 70°C, followed by 10 minutes steaming at 15 pounds pressure, gives the optimum results.

h. Four samples, 75, 76, 77, and 78, were *soaked for ½ hour, 1, 2, and 3 hours respectively at 80°C followed by 10 minutes steaming at 15 pounds pressure.* Translucent, well-parboiled kernels were obtained after 2 and 3 hours soaking at this high temperature. However, the breakage on shelling was quite high (13 and 14.6%) which might be reduced by a longer period of steaming, a higher pressure, or a more effective method of drying. The average thiamine content amounted to 2.45 μg/g.

i. Soaking at 90°C for 10, 20, 30, and 40 minutes, followed by 10 minutes steaming at 15 pounds pressure, was performed with samples numbered 79, 80, 81, and 82. The rice of the first two samples showed slightly translucent kernels. The kernels of the last two samples (81 and 82) were well parboiled, had an average of 2.22 μg/g of thiamine, and an average breakage of 5.9%.

SOAKING, BOILING, AND STEAMING: The combination of 2 hours soaking, boiling for 5 minutes, followed by steaming for 10 minutes at 15 pounds pressure, was used in sample 83 (see *d* Table I). A similar combination, using 10 minutes boiling, followed by 10 minutes steaming at 10 pounds pressure, was employed in sample 84. Both methods were failures as parboiling practices. The kernels were still opaque and up to 46% breakage was obtained during shelling. Better results were obtained with samples 85 and 86 which were soaked for 4 hours, boiled for 5 and 10 minutes, and steamed afterwards for 10 minutes at 15 pounds pressure. A low average breakage of 2.3% resulted from shelling and an average thiamine content of 2.16 μg/g was found in the milled product.

To ascertain whether vacuum application has any beneficial effect on vitamin retention, two samples, numbers 87 and 88 (see *e* Table I), were treated according to the principles of rice conversion. To the first sample, number 7, a vacuum was applied for 30 minutes which was followed by 60 pounds hydraulic pressure at 70°C for 2½ hours. The thiamine content of the milled product amounted to 2.48 μg/g. The second sample, number 88, was treated the same way except that

the vacuum application was omitted. The milled end product showed a thiamine content of 2.35 μg/g. Both samples were milled to the same degree of bran removal as all other samples. The small difference of 0.13 μg/g is considered to be insignificant and falls within the experimental error. These results are typical of 15 other experiments carried out by similar procedures.

Thiamine in Soaked Rice. The data of Table III indicate that during soaking of rough brown rice, thiamine penetrates from the outer layers of the kernel into the inner layers, and that soaking has a favorable effect on the thiamine content of the milled rice. Sample 1, without soaking, had a thiamine content of 0.44 μg/g. Sample 2, after 8 hours of soaking, had a thiamine content of 1.03 μg/g. Both samples were milled to the same degree of bran removal. Similar results were obtained in the remaining rough and brown rice samples.

Rough rice soaked in water lost small amounts (up to 4.04%) of its thiamine content in the steep water. These results indicate that only small amounts of thiamine are lost in the steep water in mills where modern commercial practices of parboiling are employed. The losses for brown rice amounted to 10.9% after 8 hours and 16% after 36 hours of soaking, and are of importance where brown rice is soaked for considerable time before it is prepared for human consumption.

Summary

A study has been made of the effect of simple, controlled, standardized methods of parboiling of rough rice on the thiamine content of the milled product (milled to approximately 10% bran removal). These methods included boiling; boiling and steaming; steaming; soaking and steaming; soaking, boiling, and steaming. The data showed that boiling alone is unsatisfactory. Of the other methods tested, the best results were obtained from the following procedures:

Boiling for 20 minutes, followed by 15 minutes steaming at 5 pounds pressure—this method produced fairly well parboiled rice, with a thiamine content of 2.13 μg/g and a breakage of 4%. The average thiamine content of the milled nonparboiled samples was 0.60 μg/g and the average breakage 3.8%.

After 15 hours soaking at room temperature (27°C), followed by 25 minutes steaming at 15 pounds pressure, the thiamine content was 2.71 μg/g and the breakage 6.1%.

After 45 hours soaking at room temperature (27°C) or a minimum of 6 hours soaking at 35°C, followed by 10 minutes steaming at 15 pounds pressure, the thiamine content was 2.20 μg/g and the average breakage 3.1%.

After 6 hours soaking at 40°C, followed by 15 minutes steaming at

15 pounds pressure (Malekized process), the thiamine content was 2.20 μg/g and the breakage 0.9%.

After a minimum of 4 hours soaking at 60°C, a minimum of 2 hours soaking at 70°C, 2 hours soaking at 80°C, at least 30 minutes soaking at 90°C, followed by 10 minutes steaming at 15 pounds pressure, the average thiamine content was 2.30 μg/g and the average breakage 4.2%.

Data are included on the effect of parboiling rough rice on breakage during shelling. It was found that prolonged boiling reduced breakage from an average of 37.8% to 27.6% and additional steaming reduced breakage further to 6.8% in boiling and steaming tests. Lowest breakage was obtained in soaking and steaming tests when soaking was followed by steaming for at least 10 minutes at 15 pounds pressure. Results are also presented on the effect of soaking of rough and brown rice on the thiamine content of the milled form and of losses of thiamine in the soaking water. After 8 hours of soaking brown rice, the thiamine content of the milled form was 1.03% to 0.44% in the milled nonsoaked sample. Losses in the soaking water for brown rice amounted to 10.9% of thiamine after 8 hours, and 16% after 36 hours of soaking. Only 4.04% thiamine was so lost in rough rice after 36 hours of soaking.

Literature Cited

Aykroyd, W. R.
1932 The effect of parboiling and milling on the anti-neuritic vitamin (B_1) and phosphate content of rice. J. Hyg. **32**: 184.

Hennessy, D. J., and Cerecedo, L. R.
1939 The determination of free and phosphorylated thiamin by a modified thiochrome assay. J. Am. Chem. Soc. **61**: 179–183.

Jack, H. W.
1923 Rice in Malaya. Malayan Agr. J. **35**.

Jones, J. W., and Taylor, J. W.
1935 Effect of parboiling rough rice on milling quality. U. S. Dept. Agr. Cir. 340.

Kik, M. C.
1945 Parboiling. Rice J. **48** (12): 6–8.
1945a Effect of milling, processing, washing, cooking and storage on thiamine, riboflavin and niacin in rice. Arkansas Agr. Expt. Sta. Bull. 458.

——, and Williams, R. R.
1945 Nutritional improvement of white rice. Bull. Natl. Research Council **112**: 1–76.

Subrahmanyan, V., Sreenivasan, A., and Das Gupta, H. P.
1938 Studies on quality in rice. I. Effect of milling on the chemical composition and commercial qualities of raw and parboiled rices. Indian J. Agr. Sci. **8**: 459–486.

STUDIES OF THE COMPOSITION OF THE WHEAT KERNEL. III. DISTRIBUTION OF ASH AND PROTEIN IN CENTRAL AND PERIPHERAL ZONES OF WHOLE KERNELS [1]

V. H. Morris, Thelma L. Alexander, and Elizabeth D. Pascoe [2]

(Received for publication July 12, 1946)

Fundamental knowledge of the relationship between the ash and protein distribution in the wheat kernel and in the mill streams is of marked importance to the flour milling industry. Adequate knowledge regarding this distribution has been difficult to obtain. In the past the principal source of information has been the circumstantial evidence secured through studies of the composition of the different flour and feed grades and the mill streams from which they are combined. Recently the authors (1945) suggested microdissection as a means of obtaining more accurate information on the distribution of ash and protein in the wheat kernel. This was accomplished by securing and analyzing material from definite parts of the kernel. The technique employed and the results obtained through its use were reported in a preliminary study of the distribution of ash and protein in center sections. In order to correlate the results obtained by dissection with those obtained in milling, it seemed desirable to apply the technique to whole kernels. This paper describes a technique for dissecting whole kernels and gives analytical data obtained in its application to several samples.

Material and Methods

The material consisted of one variety of each of three principal classes of wheat, namely, Thorne (soft red winter), Tenmarq (hard red winter), and Thatcher (hard red spring). The sample of Thorne was obtained from a single plot grown at Wooster, Ohio, in 1943. Composite samples of the other two classes were used. The Tenmarq composite was made up of equal numbers of kernels from individual samples grown at five locations in the central and southern Great Plains in 1944. The composite sample of Thatcher was made up of equal numbers of kernels from individual samples grown at seven locations in the northern Great Plains region in the same year.

The equipment and general technique used in the dissection has been described previously by Morris, Alexander, and Pascoe (1945). In this study the kernels were hydrated by exposure for 24–48 hours

[1] Cooperative investigations between the Division of Cereal Crops and Diseases, Bureau of Plant Industry, Soils, and Agricultural Engineering, Agricultural Research Administration, U. S. Department of Agriculture, and Department of Agronomy, Ohio Agricultural Experiment Station.

[2] Senior Chemist, Junior Botanist, and former Assistant Chemist, respectively, Division of Cereal Crops and Diseases at the Federal Soft Wheat Laboratory, Wooster, Ohio.

in a closed vessel containing an open dish of water and the germ, with its bran covering, was then removed with a scalpel. The germ and degermed kernels were dried for 2 hours at 130°C and weighed. In preparation for the dissection, each degermed kernel was first cut transversely at midsection and then each half kernel was cut longi-

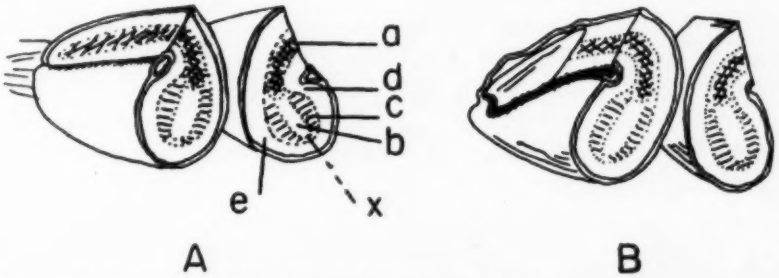


Fig. 1. Portion of kernel included in various endosperm fractions. Central zone: (*a*) "center over crease," (*b*) "center in cheek," (*c*) cheek. Peripheral zone: (*d*) endosperm next to crease, (*e*) outer endosperm, (*x*) line of separation between crease and outer portions of peripheral zone and bran fractions. (*A*) brush half of kernel; (*B*) germ half of kernel.

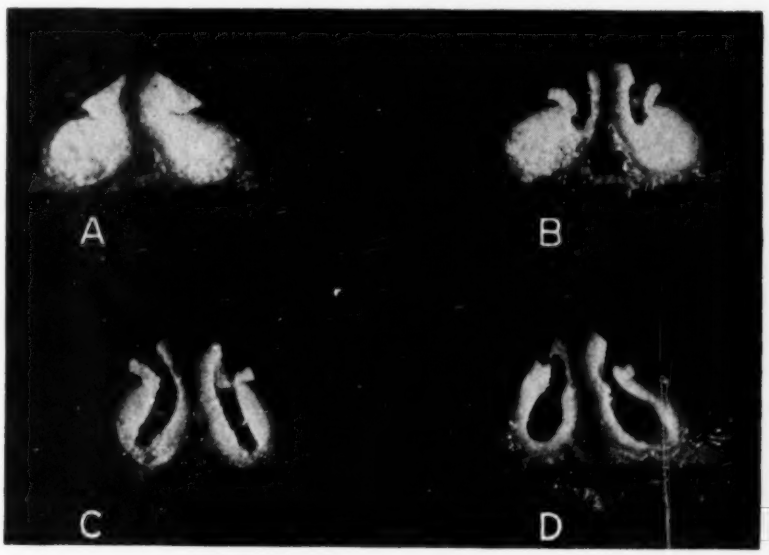


Fig. 2. Successive stages of dissection of brush halves: (*A*) mounted and ready for dissection; (*B*) after removal of center over crease; (*C*) after removal of center in cheek; (*D*) after removal of cheek. (Magnification 6 diameters.)

tudinally through the crease. The brush ends were mounted together in pairs on "Lucite" microscope slides by means of a solution of "Lucite" in chloroform. The germ ends also were mounted in the same manner.

The endosperm was separated into five fractions. The three fractions removed from the central zone were designated as "center over

crease," "center in cheek," and "cheek" endosperm. The remaining peripheral endosperm was divided into two fractions; that next to the crease was called "crease" endosperm, and the endosperm next to the outer part of the kernel was designated as "outer endosperm." The bran was separated into two fractions, "crease bran" and "outer bran." The germ with attached bran constituted another fraction. The portion of the kernel included in each fraction is illustrated by the drawings in Figure 1. The appearance of the kernel during several stages in the dissection is shown in Figure 2.

The "center over crease" and "center in cheek" were secured by drilling, using a No. 79 or 80 wire-gauge twist drill, whereas the "cheek" was obtained by drilling with a No. ½ dental burr. An incision was then made at the base of the cheek dividing the remainder of the kernel into two parts—"crease" and "outer"; the endosperm of each part was separated from the bran by cutting and scraping with a sharp scalpel. The dissected fractions were dried for 1 hour at 135°C, weighed and ground in a micro-Wiley mill. Protein was determined on 6–12 mg subsamples by a micro-Kjeldahl procedure. Ash content was determined by ashing the remainder of each fraction in a semimicro platinum dish, the weighings being made on a micro-analytical balance.

Results

The dissection and analytical data are presented in Table I. For convenience in discussion, the quantities of dry matter, ash, and protein in certain fractions have been summed or averaged. The data for the center over crease and center in cheek are referred to as "center endosperm"; these fractions combined with that from the cheeks are referred to as "central zone." The crease and outer endosperm are referred to as "peripheral zone" and the crease and outer bran as "total bran," etc. The data obtained by calculation rather than by analysis are shown in italics in Table I.

One of the difficulties encountered was in obtaining a reasonable recovery of the original undissected kernels. In this study losses of 14, 13, and 22% of dry matter occurred with the soft winter, hard winter, and hard spring varieties, respectively. These undoubtedly represent fine particles lost to the air in drilling as well as losses in the handling of dissected material. Unfortunately there was no way of ascertaining how this loss should be apportioned among the several fractions, but the high proportion of bran and germ shown in Table I indicates that the losses were chiefly in the endosperm fractions.

Ash. In all three varieties the lowest ash concentration was found in one of the two center endosperm fractions. The ash content of these

TABLE I

COMPOSITION OF FRACTIONS OF WHEAT KERNELS [1]

Parts of kernel	Number of kernels dissected			Kernel composition								
				Dry weight per 100 kernels			Ash in each fraction or part			Protein in each fraction or part		
	Thorne	Tenmarq	Thatcher	Thorne	Tenmarq	Thatcher	Thorne	Tenmarq	Thatcher	Thorne	Tenmarq	Thatcher
				g	g	g	%	%	%	%	%	%
Endosperm												
Center over crease	200	199	180	0.214	0.233	0.193	0.33	0.42	0.38	7.1	9.5	8.3
Center in cheek	200	199	180	.355	.316	.291	.29	.42	.38	6.2	8.5	8.4
Cheek	200	199	195	.320	.244	.266	.32	.47	.44	7.4	9.5	10.2
Crease	75	75	75	.562	.591	.477	.63	.55	.57	11.6	12.6	13.9
Outer	75	75	75	.921	.844	.745	.54	.56	.53	10.5	13.7	15.1
Center				*.569*	*.549*	*.484*	*.31*	*.42*	*.38*	*6.5*	*8.9*	*8.4*
Central zone				*.888*	*.773*	*.750*	*.31*	*.43*	*.40*	*6.9*	*9.0*	*9.0*
Peripheral zone				*1.483*	*1.435*	*1.222*	*.57*	*.56*	*.55*	*10.9*	*13.2*	*14.5*
Total endosperm				*2.371*	*2.208*	*1.972*	*.48*	*.51*	*.49*	*9.4*	*11.7*	*12.5*
Bran												
Crease	75	75	75	.377	.319	.295	5.31	4.46	4.64	12.9	15.1	14.4
Outer	75	75	75	.460	.352	.329	6.72	6.21	6.76	13.1	14.9	14.6
Germ [2]	200	200	200	.149	.109	.119	5.87	5.38	5.35	25.5	26.8	24.3
Total bran and germ				*.986*	*.780*	*.743*	*6.05*	*5.38*	*5.68*	*14.9*	*16.5*	*16.2*
Total dissected kernel				*3.357*	*2.988*	*2.715*	*2.12*	*1.79*	*1.91*	*11.0*	*13.0*	*13.5*
Wheat kernel												
Degermed kernel				3.763	3.322	3.386	1.65	1.40	1.56	10.0	11.5	12.6
Germ [2]				.149	.109	.119	5.87	5.38	5.35	25.5	26.8	24.3
Whole kernel				*3.912*	*3.431*	*3.505*	*1.81*	*1.53*	*1.69*	*10.6*	*12.0*	*13.1*

[1] Data on a 14% moisture basis.
[2] Includes attached bran.

two fractions was not significantly different within the variety for the hard wheat, but in the soft wheat the "center over the crease" was slightly higher than the "center in the cheek." The "cheek" fraction was higher in ash content than the "center in the cheek" for all three varieties, the smallest difference occurring in the soft wheat. While this may be regarded as evidence of an increasing gradient in concentration from the center of the endosperm to the bran coat, the gradient does not appear very steep in this part of the endosperm. The ash content of the two peripheral endosperm fractions—crease and outer—was considerably greater than the central zone fractions for all three varieties. The average ash content of the peripheral zone exceeded that of the central zone by 0.26, 0.13, and 0.15% for Thorne, Tenmarq, and Thatcher respectively. Although only two principal endosperm zones are represented in the dissections, the results corroborate those previously obtained by Morris, Alexander, and Pascoe (1945) in that they indicate the existence of major differences in concentration of ash in various parts of the endosperm. While it will be necessary to analyze a number of samples of the different classes of wheat to establish evidence of fundamental difference between them, the greater spread in concentration between the central and peripheral zones in the Thorne sample would seem to have some significance since in commercial milling it is common practice to obtain lower ash patent flours from soft wheat.

The ash content of the endosperm as a whole (i.e., of the five endosperm fractions) was lowest for Thorne (0.48%), highest for Tenmarq (0.51%), and intermediate for Thatcher (0.49%). These figures all appear somewhat high considering that straight grade flours containing considerably lower ash contents are commonly milled from commercial lots of these wheats. This point is considered further in a later part of the paper.

Protein. The lowest concentrations of protein in all three varieties were found in the center endosperm fractions. The "cheek" fraction exceeded the "center in the cheek," the difference ranging from 1.0 to 1.8% in all three varieties, suggesting the existence of a slightly increasing gradient in protein content as was found also for ash. In general, however, the data indicate that differences in the composition of the central zone within a variety are not marked with respect to protein.

The protein content of the two fractions constituting the peripheral zone was considerably higher than that of the central zone in all three varieties. The increase amounted to 60, 47, and 61% for Thorne, Tenmarq, and Thatcher, respectively.

Comparison of Dissected Fractions and Experimental Mill Streams.

It is a common observation that major differences in ash and protein content such as these found in dissected fractions also occur in flour streams obtained in commercial and experimental milling. The principal difference between the two processes is that in dissection it is possible to obtain endosperm fractions free of bran; whereas, in milling, the flour streams may be contaminated with a small quantity of pulverized bran and germ particles. This is especially true of flour streams from the tail end of the mill. To determine if the range in composition of flour streams obtained by milling agrees reasonably well with that found in pure endosperm obtained by dissection, 2,500 g of the Thorne sample were milled on a Buhler experimental mill. The yield obtained by dissection and through mill streams and the analytical data are presented in Table II.

TABLE II

COMPARATIVE YIELD AND COMPOSITION OF FRACTIONS FROM THORNE SOFT WINTER WHEAT OBTAINED BY DISSECTION AND BY MILLING [1]

Dissection fractions	Dry matter proportion of total dissected [2]	Ash	Protein	Buhler mill streams	Dry matter proportion of total milled	Ash	Protein
		%	%			%	%
Endosperm							
Center over crease	5.5	0.33	7.1	1st break flour	9.4	0.29	7.1
Center in cheek	9.1	.29	6.2				
Cheek	8.2	.32	7.4	2nd break flour	9.8	.34	9.6
Crease	14.4	.63	11.6	3rd break flour	3.6	.60	11.8
Outer	23.5	.54	10.5	1st reduction flour	21.5	.36	9.1
Center	*14.5*	*.31*	*6.5*				
Central zone	*22.7*	*.31*	*6.9*	2nd reduction flour	11.3	.47	9.9
Peripheral zone	*37.9*	*.57*	*10.9*	3rd reduction flour	8.0	.61	11.0
Total endosperm	*60.6*	*.18*	*9.4*	*Total flour*	*63.6*	*.11*	*9.4*
Bran							
Crease	9.6	5.31	12.9	Bran	20.6	5.91	14.3
Outer	11.8	6.72	13.1				
Germ [3]	3.8	5.87	25.5	Shorts	10.8	2.71	13.5
Total bran and germ	*25.2*	*6.05*	*14.9*	*Total bran and shorts*	*31.4*	*4.84*	*14.0*
Total dissected kernel	*85.8*			*Total milled products*	*95.0*		

[1] Data on a 14% moisture basis.
[2] Percentage of whole undissected kernel.
[3] Includes attached bran.

The lowest concentration of ash, which was found in the first break flour (0.29%), was similar to that of the lowest ash dissected fraction—"center in the cheek"; in fact it agrees, within the limits of analytical error, with the ash content of the whole central zone (0.31%). The second break and first reduction flours were somewhat higher in ash content than central zone fractions. These two streams together with the first break represented 40.7% extraction with an ash content of 0.34%, compared with 22.7% extraction and 0.31% ash for the central zone obtained by dissection. The high ash flour streams,

third break (0.60%) and the third reduction (0.61%), contained about the same quantity of ash as the highest dissected fraction—crease endosperm (0.63%). Similarly, the lowest-protein flour stream (first break, 7.1%) was about equal in concentration to the average of the central zone (6.9%), and the high protein flour streams (third break, 11.8%, third reduction, 11.0%) were about equal in content to the two peripheral endosperm fractions (11.6 and 10.5).

While it is recognized that there is a distinct limit to which experimental milling can be interpreted in terms of the commercial process, the results of this study suggest that the relatively high ash of certain flour streams may be accounted for by the ash content of peripheral endosperm. The results provide little evidence that incorporation of pulverized bran was an important factor in determining the ash content of any of the flour streams. In fact the trend is in the opposite direction since the ash content of the total dissected endosperm of the Thorne sample was 0.48% compared with 0.41% for the total flour obtained by milling. Further evidence of a discrepancy between dissected and milled results was found in the data obtained on Tenmarq and Thatcher. Individual samples of Tenmarq from each of the five locations and of Thatcher from each of the seven locations represented in the respective composites for dissection were milled separately on the Buhler mill and the results for each variety were then averaged. The flour yield, ash, and protein obtained from dissection and from milling of the three varieties, including Thorne, were as shown in Table III.

TABLE III

DISSECTED ENDOSPERM VS. MILL STREAM

Variety	Total dissected endosperm			Straight grade flour		
	Yield	Ash	Protein	Yield	Ash	Protein
Thorne	60.6	0.48	9.4	66.8	0.41	9.4
Tenmarq	64.3	.51	11.7	68.0	.43	11.8
Thatcher	56.3	.49	12.5	62.4	.46	12.2

The difference in ash content between dissected endosperm and straight grade flour is particularly wide with the Thorne and Tenmarq samples, for which the most satisfactory dissection and milling results were obtained. Whether the variations are due to experimental error or to differences inherent in the two processes cannot be determined with certainty with the data at hand. It is possible that the dissected endosperm ash value is relatively too high because of significant losses of material from the lower ash central zone endosperm during the

process of dissection. The other possibility is that the shorts produced in the milling operation may carry appreciable quantities of flour containing high ash peripheral endosperm, which would result in lowering the ash content of the straight flour. It may be noted, however, that the discrepancy does not appear in the protein determinations, there being excellent agreement in regard to this constituent between dissected endosperm and straight grade flour in all three varieties.

Summary

A microdissection technique previously developed was employed to separate whole wheat kernels into fractions as follows: (1) five separations by zones of pure endosperm, (2) two of bran, and (3) one of the germ (including attached bran). These were obtained from three classes of wheat—soft red winter (Thorne), hard red winter (Tenmarq), and hard red spring (Thatcher). The Thorne sample came from a single location, whereas the hard wheat samples were composites representing a number of locations all grown in 1944.

In all three varieties the lowest concentration of ash was found in one of the "center" endosperm fractions. Likewise in all three varieties a slight but consistent increase in ash content of the "cheek endosperm" fraction was found as compared with the "center in the cheek." The ash content of the peripheral endosperm fractions was considerably greater than any of the central zone fractions, although the magnitude of the difference varied considerably among the three varieties. In general, similar results were obtained with respect to the distribution of protein in the various endosperm fractions. These facts furnish considerable evidence of an increasing gradient in ash and protein content from the center of the endosperm outward.

A comparison of the ash and protein contents of the dissected fractions with those of Buhler flour mill streams of Thorne soft wheat showed that the lowest-ash dissected fraction (center in cheek, 0.29%) was identical with that of the lowest-ash flour stream (first break, 0.29%). The highest-ash flour streams (third break, 0.60%, and third reduction, 0.61%) were slightly lower than the highest-ash dissected fraction (crease endosperm, 0.63%).

The ash content of total dissected endosperm in all three varieties was somewhat higher than that of the straight grade flours. The protein contents of total dissected endosperm and straight grade flours, however, were in close agreement.

Literature Cited

Morris, V. H., Alexander, T. L., and Pascoe, E. D.
1945 Studies of the composition of the wheat kernel. I. Distribution of ash and protein in center sections. Cereal Chem. 22: 351–361.

PHOTOMICROGRAPHIC STUDIES OF WHEAT STARCH. II. AMYLOLYTIC ENZYMES AND THE AMYLASE INHIBITOR OF THE DEVELOPING WHEAT KERNEL [1]

R. M. SANDSTEDT and O. C. BECKORD

Department of Agricultural Chemistry, Nebraska Agricultural Experiment Station, Lincoln, Nebraska

(Received for publication June 26, 1946)

This paper supplements the one concerning the development of starch granules in wheat (Sandstedt, 1946) with photomicrographs and chemical data concerning amylase activity in the developing wheat kernel.

Relationships between starch deposition and amylase activity in growing plants have not been extensively studied. Until recently methods for quantitatively differentiating the actions of alpha- and beta-amylase were not available. Teller (1938) used photomicrographs of immature starch granules to support his hypothesis that the growth of starch granules was due to the synthetic action of amylases. Bernstein (1943) attempted to relate amylase activity to the rate of starch formation in the developing *maize* kernel. He concluded that the amylases were not active as synthetic agents. Chrzaszcz and Janicki (1936) reported liquefying, dextrinizing, and saccharifying action of enzymes from the grain of wheat, oats, barley, and rye immediately after blossoming and showed that the liquefying and dextrinizing functions gradually disappear, with a partial loss of saccharifying function, as the grain ripens. Sandstedt (1946), while studying the development of starch in wheat, noticed that the starch deposited in the pericarp was digested during the development of the kernel.

Materials and Methods

Kernels of wheat were selected from greenhouse-grown plants at several stages of development. The stages of kernel growth referred to in this paper as *A*, *B*, *C*, etc., correspond to those shown in Figure 1 (reprinted from Sandstedt, 1946). The stage indicated by *G*+ represents about four days later than *G*.

Preparation of Samples. At stage *D* the kernel was sufficiently developed to allow a separation of the embryo, pericarp, and endosperm.[2] Each tissue was washed with water as soon as it had been isolated, since in dissection there was possibility of considerable contamination of one part by the juices from another. Such washing

[1] Published with the approval of the Director as paper No. 396, Journal Series, Nebraska Agricultural Experiment Station.

[2] We are indebted to James Fleming for the dissection and preparation of the samples.

necessarily introduced some error in the quantitative determinations of enzyme action.

The immature whole kernels and the dissected tissues were dried at room temperature in a current of air from an electric fan. Sufficient material was prepared to give at least 50 mg of dried sample for each analysis.

The endosperm was prepared for analysis by grinding in a mortar. The pericarp and whole kernels were ground with glass. A 50 mg (air-dried) sample was extracted with 5 ml of a 0.2% $CaCl_2$ solution for one hour at 30°C, and then centrifuged.

The dissection of the wheat kernel, especially in the early stages of growth, was tedious and time consuming: at stage *D* a dry endo-

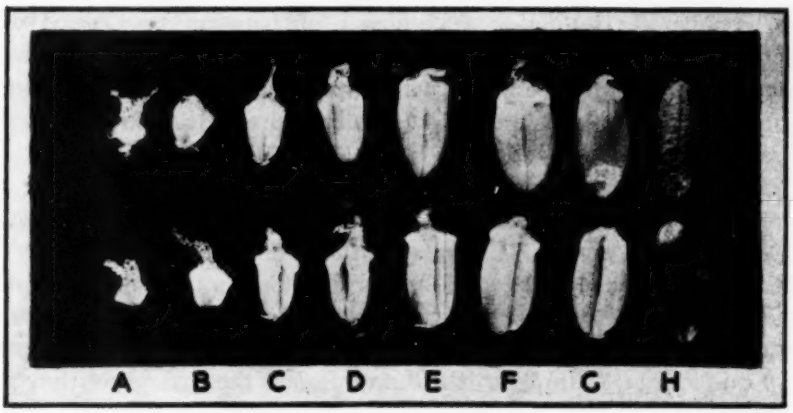


Fig. 1. Wheat kernels showing change in size and form during development (×2.5).
A. Unfertilized ovary.
B–G. Kernels approximately representative of development to be expected at 2-day intervals.
H. Mature dry kernel.

sperm weighed approximately 1 mg. Accordingly, the enzyme methods were modified for the use of small quantities of material.

Alpha-Amylase Determination. The method of Sandstedt, Kneen, and Blish (1939) was modified for use of small quantities of material as follows: 4 ml of buffered alpha-amylodextrin substrate was treated with 2 ml of enzyme extract (equivalent to 20 mg of dry tissue). The reaction was carried out in 15 ml tapered centrifuge tubes in the 30°C water bath. One-half ml of iodine-potassium iodide solution ("solution B") was measured into ½ dram vials. Two drops (0.1 ml) of the reaction mixture was added to the 0.5 ml of iodine solution to follow the progress of the reaction.

For the micro amounts of alpha-amylase found in the endosperm, the "overnight method" (Kneen, Sandstedt, and Hollenbeck, 1943) was used wherein 1 ml extract was allowed to act on 4 ml substrate

for 18 hours. The reaction was then completed by the addition of 1 ml of an extract of wheat malt of known activity.

The unit of alpha-amylase activity as defined by Sandstedt, Kneen, and Blish (1939) is used in this paper—the amount of alpha-amylase which (under the influence of an excess of beta-amylase) will dextrinize 1 g of boiled soluble starch in one hour at 30°C (usually expressed as units per gram of sample).

Beta-Amylase Determination. The method of Kneen and Sandstedt (1941) was modified for use of small quantities of material as follows: 1 ml of tissue extract was added to 8 ml of 2% starch substrate and 3 ml of water. The reaction was carried out in a 50-ml Erlenmeyer flask at 30°C. At the end of the 15-minute reaction period, 8 ml of 1% H_2SO_4 solution were added. A 5-ml aliquot was taken for ferricyanide reduction. The unit of beta-amylase activity as defined by Kneen and Sandstedt (1941) is used in this paper—the amount of beta-amylase which will convert 1 g of boiled soluble starch to maltose in one hour at 30°C (usually expressed as units per gram of sample).

Photomicrographs. The technique and equipment for taking the photomicrographs was the same as that used by Sandstedt (1946).

Experimental Error. Clean, quantitative separation of tissues was difficult in the early stages of kernel development. The endosperm tended to cling to the pericarp, and washing the dissected tissues entailed inevitable losses. Therefore experimental errors were probably quite large. However, the data do serve their purpose; they give a rough quantitative measure of alpha- and beta-amylase in the tissues and indicate the changes which occur during the development of the kernel.

Results and Discussion

Amylolytic Action in the Immature Wheat Kernel. Teller (1938) hypothesized that starch granule growth was due to amylase action, i.e., that the amylases were active as *synthetic* agents in the tissues in which starch was being deposited. The digestion of the pericarp starch during the growth of the kernel shows that an enzyme system capable of attacking the starch granule is present in this tissue (Sandstedt, 1946).

In preliminary trials, wheat kernels at the several stages of development were autolyzed for 24 to 48 hours (immersed in water, 0.2 *M* acetate buffer at pH 4.7, 1:1,000 Roccal [3] solution, or buffer and Roccal solution), and then crushed and examined. In all tests the

[3] Roccal is a detergent antiseptic obtained from Winthrop Chemical Company, New York City. It was used in these cases to check against the possibility of the starch digestion being due to bacteria or fungal growth during the extended autolysis. Roccal should not be indiscriminately used because under some conditions it has an effect on enzyme action.

pericarp starch had been extensively digested, whereas the endosperm starch showed only slight digestion. However, if the kernels of wheat were crushed before autolysis, the endosperm starch was more rapidly digested than the pericarp starch. Such evidence suggested that the enzymes capable of digesting native starch granules were present and active in the pericarp, perhaps absent in the endosperm, and that immature endosperm starch was more readily attacked by enzymes than was pericarp starch.

The pericarp (with the nucellus) and the endosperm were dissected from a series of kernels of wheat representing about three- or four-day intervals during the first 18 days of kernel development. Each pericarp and each endosperm was allowed to autolyze (in buffered Roccal solution) for 24 hours, was then crushed, and the amount of starch digestion observed. Considerable starch digestion took place during the pericarp autolysis, whereas no evidence of enzyme action could be seen after endosperm autolysis. Apparently the active enzyme system capable of attacking starch granules was present only in the pericarp.

The above procedure was not satisfactory for photomicrographic illustration since the progressive digestive action on a particular field of starch granules could not be followed. Also, pericarp starch granules are not satisfactory for photomicrographic studies of enzyme action because of their small size and previous exposure to enzyme action. Hence, each endosperm was crushed in a drop of 0.2 *M* acetate buffer (pH 4.7) on a glass slide and then sealed under a cover glass; each pericarp also was treated in this way except that *washed* [4] endosperm starch was added as additional substrate. This procedure permitted photomicrographs to be taken of a satisfactory field of starch granules before, as well as after, intervals of autolysis.

For photomicrographic illustration, the action obtained from the pericarp and endosperm dissected from a kernel harvested the 10th day of development was chosen as typical of the entire period of development. Figure 2 shows endosperm and pericarp starch granules in the dispersion made from the pericarp: *A* at the beginning of the digestion, *B* and *C* after 5 hours and 24 hours at 30°C. The pericarp granules were subject to Brownian movement; accordingly, many of those shown in *A* had moved and cannot be identified in *B*; few can be identified in *C*. It is evident that the pericarp contained an enzyme system capable of attacking immature starch granules. Some of the pericarp granules were quite resistant to the enzyme action, whereas the endosperm starch was completely digested in 24 hours.

[4] Isolated endosperms were crushed in water and the starch so obtained was washed by suspension, settling, and decantation. Washed starch was used in order to avoid contaminating the pericarp digest with enzymes and inhibitors from the endosperm.

The pericarp enzyme system was less active in those preparations representing late stages of pericarp development. By the 14th to 16th day of development the pericarp had digested most of its own starch and had lost considerable of its amylolytic activity.

The shrinkage, as distinct from erosion, in the immature endosperm granules during the early stages of enzyme action, i.e., the difference in diameter of the granules before and after a period of digestion, as shown in Figure 1 *A* and *B*, is of interest. Such shrinkage is not evident during the action of enzymes on mature native granules, whereas it is marked during the action of beta-amylase on partially gelatinized granules (unpublished data).

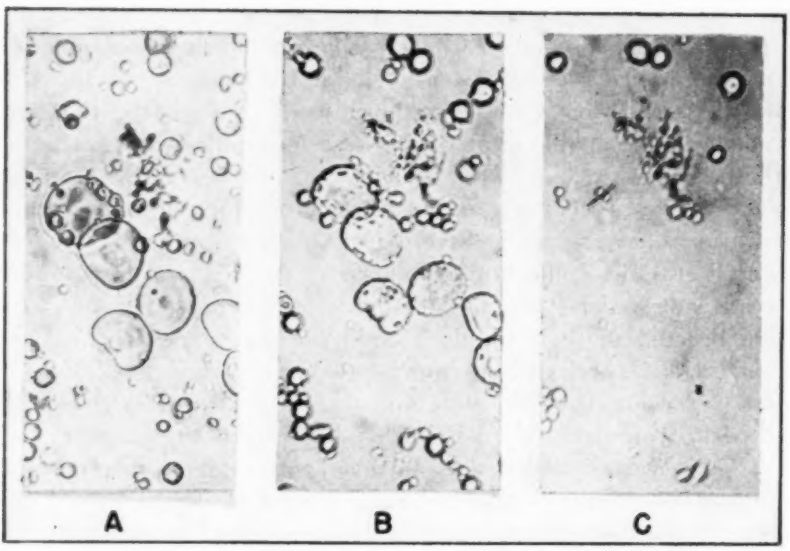


Fig. 2. Action of pericarp enzymes on immature pericarp and endosperm starch granules (×600).
A. Starch at initiation of digestion.
B. After 5 hours.
C. After 24 hours.

Figure 3 illustrates the absence of autolytic starch granule degradation in the crushed endosperm during a 72-hour period. If enzymes capable of digesting native starch granules were present in the endosperm, they were apparently in an inactive form. This does not preclude the presence of beta-amylase, which is unable to attack undamaged immature starch granules. (It is shown later in this paper that beta-amylase is present in the endosperm.)

The absence of starch digestion in the crushed endosperm was not due to the use of a pH other than the optimum, since the occurrence of starch granule digestion in the crushed pericarp and its absence

in the crushed endosperm were evident, whether the dispersions were at the natural pH of the crushed tissue, buffered at pH 4.7 with acetate, or at pH 6.0 with phosphate.

The Occurrence of an Amylase Inhibitor. During the course of this work it became evident that the resistance of starch granules to amylase action increased with maturity. The preliminary procedure used for studying resistance to amylase action was to crush the entire kernel of wheat in a drop of diluted buffered saliva and to follow the action of the salivary enzyme by microscopic observation. The salivary amylase had a relatively rapid action on the immature starch; the rate of action decreased with increasing maturity of the starch. However, at a stage of development corresponding about to the attainment of full kernel length, salivary enzyme action ceased to take place. After this stage of development, no more starch digestion took place

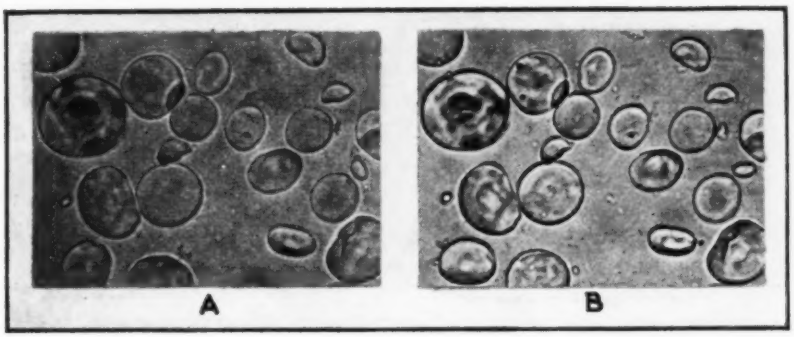


Fig. 3. Inability of the immature crushed endosperm to digest its own starch granules (×600).
A. At the initiation of digestion.
B. After 72 hours digestion.

in the presence of saliva than could be accounted for by the action of the pericarp enzymes alone. Yet if washed starch were treated with saliva, normal digestion occurred. It was then found that extracts of wheat flour and even of wheat malt would inhibit salivary amylase action. Apparently an inhibitor of certain types of amylases was developed in the growing wheat kernel at about the time that full length was attained.

Since the pericarp and endosperm differ in amylase activity, it was of interest to investigate the possible presence of the amylase inhibitor in each of these tissues. Endosperms and pericarps were dissected from kernels at the full length stage. Each endosperm was crushed in a drop of buffered saliva on a glass slide, sealed beneath a cover glass, and placed at 30°C for digestion. This procedure was repeated with the pericarps, but since at this stage an undried endosperm

weighed about five times as much as an undried pericarp, five pericarps were used, together with about 0.5 mg of washed endosperm starch, in each drop of buffered saliva. Check slides were prepared using buffer solutions with no saliva and using 0.5 mg of *washed* endosperm starch [4] alone in buffered saliva.

Typical photomicrographs obtained after a 6-hour digestion are shown in Figure 4. No evidence of enzyme action was shown in the presence of the crushed endosperms (Figure 3 *A*), which indicates an inactivation of the salivary amylase system; as before noted, there was no active enzyme system in the endosperm itself capable of attacking starch granules.

On the other hand, the salivary enzymes were active in the presence of the crushed pericarp (Figure 4 *B*). That most of the action shown in Figure 3 *B* was due to salivary amylase and little due to the peri-

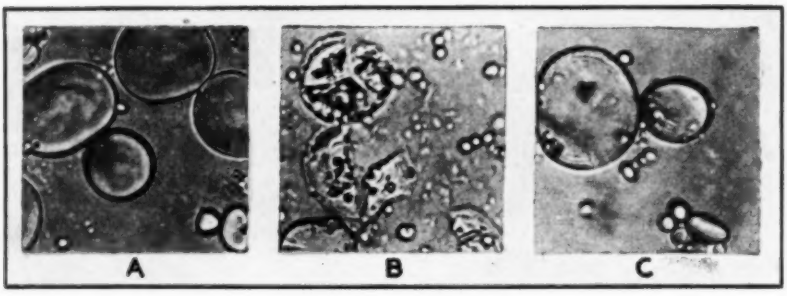


Fig. 4. Salivary amylase inhibitor as found in the endosperm and pericarp on the 16th day of kernel development.
All photomicrographs taken after 6 hours digestion (×600).
A. Showing complete inhibition of salivary digestion in suspension of crushed endosperm.
B. Action of salivary and pericarp amylases on washed endosperm starch in the presence of the crushed pericarp—no inhibition of salivary amylase by the crushed pericarp.
C. Pericarp amylase alone on washed endosperm starch in the presence of crushed pericarp.

carp enzyme system is shown by comparison with Figure 4 *C* which shows the action of the enzymes of the crushed pericarp on washed endosperm starch. Apparently a salivary amylase inhibitor was present in the endosperm but not in the pericarp. This is substantiation of the work of Kneen and Sandstedt (1946); they showed that wheat bran extracts caused little inhibition of salivary or bacterial amylases.

Correlation of Microscopic Observations with Chemical Analysis. The data of Figure 5 correlating the concentration of alpha-amylase in the kernel and in the kernel tissues with the age of the kernel indicate that the alpha-amylase was practically all in the pericarp with only traces in the endosperm and that the concentration in the pericarp decreased with development.

In accordance with the work reported by Chrzaszcz and Janicki (1936), there was a steady decrease in alpha-amylase concentration

in the whole kernel during the growth of the kernel. They, however, could partially restore the original amylolytic power of the grain (but only up to a certain stage of development) by the use of "eleuto-substances" (peptone or weak solutions of salt). They explain the fact that restoration was only partial, i.e., their inability to obtain as high values during the later stages of kernel development as during the early stages, by hypothesizing an excessive accumulation of "sisto-substances" which bind and thus inactivate the amylase to such an extent that the inactivation could not be nullified by "eleuto-substances."

The data of Figure 5 indicate that the decrease in alpha-amylase concentration in the whole kernel during development was due to two

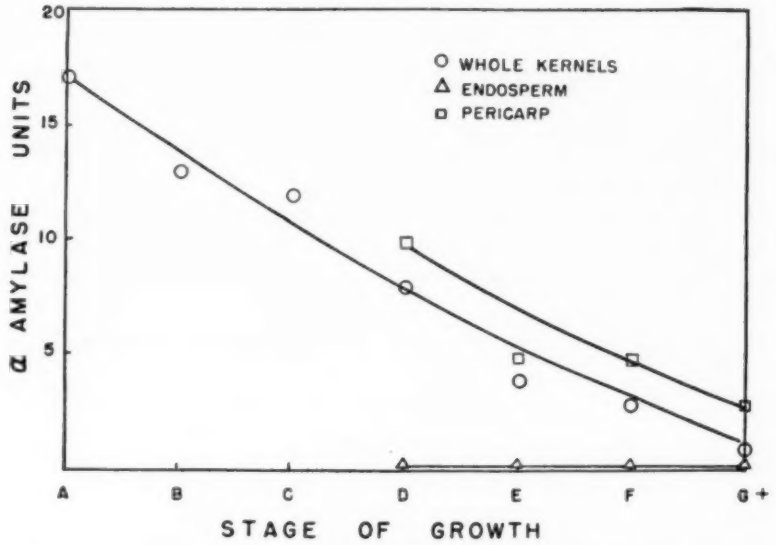


Fig. 5. Relation of stage of wheat kernel development to concentration of alpha-amylase. Alpha-amylase expressed as units per gram of air-dried tissue.

factors: 1. A decrease in the content of active alpha-amylase in the pericarp tissue. We were unable to increase the alpha-amylase activity of extracts from tissues of immature kernels by the use of the "eleuto-substances" mentioned by Chrzaszcz and Janicki. 2. The rapid growth of the endosperm (containing only traces of alpha-amylase) with a consequent decrease in the proportion of the kernel occupied by the tissues rich in alpha-amylase.

Figure 6, showing the changes in alpha-amylase expressed on a per kernel basis, indicates that there was an increase of alpha-amylase in the kernel during the first week of development (to stage *D*) after which there was a decrease. The increase occurred during the period

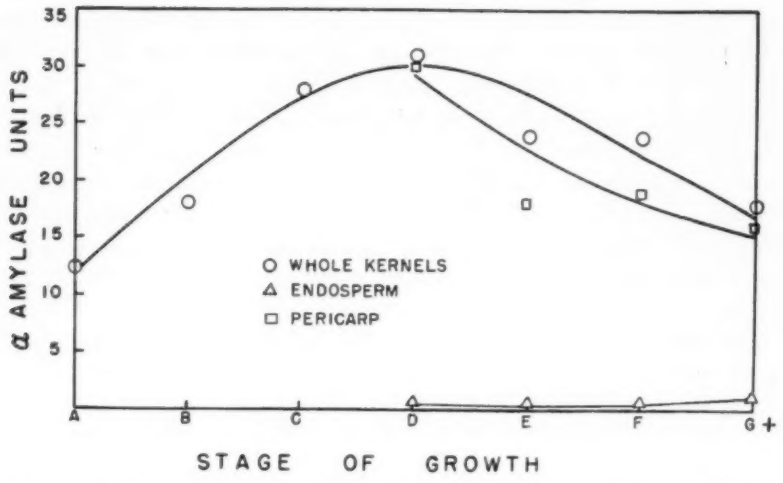


Fig. 6. Relation of stage of kernel development to total alpha-amylase content of the kernel and of the kernel tissues. Alpha-amylase expressed as units per 1,000 kernels.

of rapid growth of the pericarp. The alpha-amylase concentration in the pericarp was decreasing during this period (Figure 5), but the increase in pericarp tissue was rapid enough to cause an increase in total alpha-amylase. After stage *D* the increase in pericarp tissue was too slow to overbalance the decrease in concentration of alpha-amylase.

Figure 7 showing beta-amylase on a per gram basis indicates a

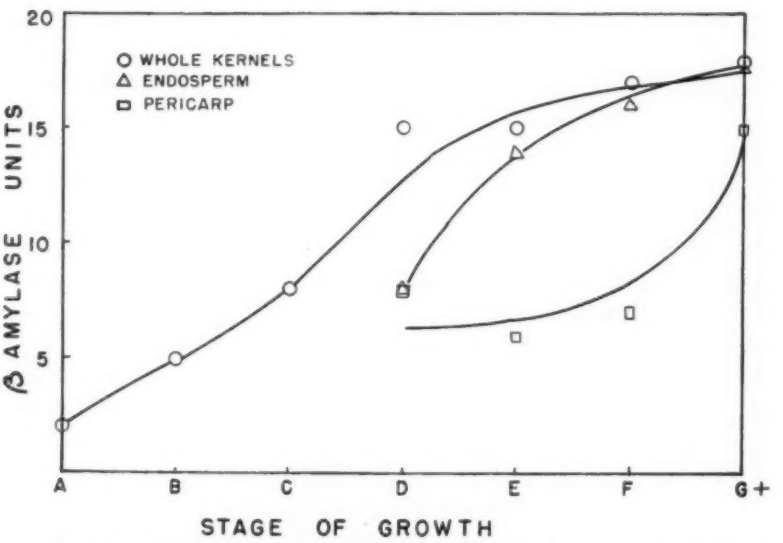


Fig. 7. The relation of stage of kernel development to concentration of beta-amylase. Beta-amylase expressed as units per gram of air-dried tissue.

rapid increase in concentration of beta-amylase in the whole kernel for about the first six days of growth, after which the concentration was almost constant. The concentration of beta-amylase in the endosperm increased rapidly during the period of change in endosperm character, i.e., from a tissue containing little starch to a starchy tissue (stages *D* to *G*). Chrzaszcz and Janicki (1936) noted a decrease in saccharifying power during the later stages of the development of the kernel. This decrease, however, was in saccharification due to the combined action of the alpha- and beta-amylases and so may have been due to the decrease in alpha-amylase (Figure 5). The pericarp also contained considerable amounts of beta-amylase.

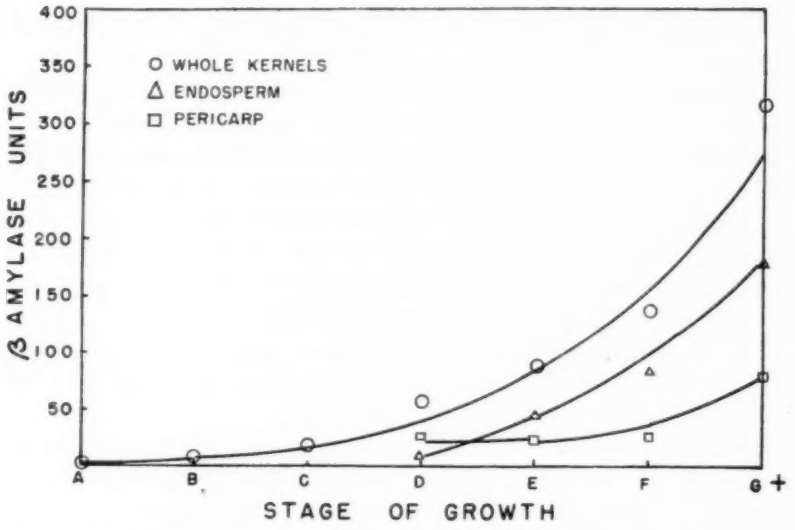


Fig. 8. Relation of stage of kernel development to total beta-amylase content of the kernel and of the kernel tissues. Beta-amylase expressed as units per 1,000 kernels.

Figure 8, showing the units of beta-amylase on a per kernel basis, indicates an increase in the total beta-amylase in the kernel with growth corresponding to the growth of the endosperm.

Embryo. Determinations of amylase activity of the embryo are not reported, as sufficient samples were not obtained to make reliable determinations. Such results as were obtained indicated that beta-amylase was present in smaller amounts in the embryo than in the endosperm and that alpha-amylase was present but in smaller amounts (on a per gram basis) than in the pericarp.

Effect of Proteolytic Enzyme Action on the Extraction of Amylases of the Immature Kernel. Eighteen-hour extractions at 30°C were made on samples of endosperm at stage *G* and whole kernel at stage *D* using

a solution containing 0.2% $CaCl_2$ and 10 mg % ficin.[5] No appreciable increase in either alpha- or beta-amylase activity was observed in these extracts, as compared to those made without the use of the proteolytic enzyme. Apparently the decrease in alpha-amylase during the growth of the kernel was not due to a tie-up of the amylase similar to that of beta-amylase in the mature kernel. Chrzaszcz and Janicki (1936) could not increase dextrinizing and liquefying powers by the action of papain.

Summary

Photomicrographs are shown indicating the presence of an amylase system in the pericarp of immature wheat capable of digesting native starch granules. However, no active enzyme capable of attacking native granules could be found in the endosperm at any stage of its development.

Chemical determinations of amylase activity on extracts of pericarp and of endosperm tissue from wheat at several stages during its development substantiated the data obtained by microscopic observation, indicating the presence of alpha-amylase in the pericarp with only traces in the endosperm. The concentration of alpha-amylase decreased during the growth of the kernel, whereas the total alpha-amylase in the kernel increased during the first week of development (period of most rapid growth of pericarp tissue) and then decreased.

The concentration as well as the total quantity of beta-amylase increased during the growth of the kernel, most of the increase being due to the growth of the endosperm.

An amylase inhibitor (which had no effect on the natural wheat or malt amylases but which was effective in inhibiting salivary amylase) appeared in the endosperm at approximately the time that the immature kernel reached full length. This inhibitor was not found in the pericarp tissue.

Literature Cited

Bernstein, L.
1943 Amylases and carbohydrates in developing maize endosperm. Am. J. Botany **30**: 517–526.

Chrzaszcz, T., and Janicki, J.
1936 Amylase during the growth and ripening of grains. Biochem. J. **30**: 1298–1302.

Kneen, E., and Sandstedt, R. M.
1941 Beta-amylase activity and its determination in germinated and ungerminated cereals. Cereal Chem. **18**: 237–252.
1946 Distribution and general properties of an amylase inhibitor in cereals. Arch. Biochem. **9**: 235–249.

——, ——, and Hollenbeck, C. M.
1943 The differential stability of the malt amylases—separation of the alpha and beta components. Cereal Chem. **20**: 399–423.

[5] A proteolytic enzyme of the papain type which is approximately 20 times as active as papain Merck and Company, Inc., Rahway, New Jersey.

Sandstedt, R. M.
1946 Photomicrographic studies of wheat starch. I. Development of the starch granules. Cereal Chem. **23**: 337–359.
——, Kneen, E., and Blish, M. J.
1939 A standardized Wohlgemuth procedure for alpha-amylase activity. Cereal Chem. **16**: 712–723.
Teller, G. L.
1938 Plant diastase in evidence as to the formation and structure of starch granules. Plant Physiol. **13**: 227–240.

SOME OBSERVATIONS ON THE VISUAL COLOR OF TUBULAR AND DISC MACARONI

L. D. SIBBITT and R. H. HARRIS

North Dakota Agricultural Experiment Station, Fargo, North Dakota

(Received for publication May 2, 1946)

Fifield, Smith, and Hayes (1937) described a method for producing micro macaroni discs for assessing macaroni-making quality and color characteristics. Cunningham and Anderson (1943) pointed out that difficulty was experienced in obtaining reliable results by the above method and they therefore developed a modified procedure. These workers also studied the influences of variations in processing conditions on the opacity of the discs (Cunningham and Anderson, 1943a, 1943b). Smith, Harris, Jesperson, and Sibbitt (1946), employing equipment similar to that described by Fifield *et al.* (1937), also used a modified procedure of the original technique for making the discs. Smith and associates showed quite clearly the effect of degree and duration of pressing on air bubble size and number, and light transmission of the discs.

Fifield *et al.* (1937) made comparisons of the discs with tubular macaroni. An N-A colorimeter equipped with four color discs was used, but only the yellow component was selected as an index of comparison. Satisfactory results were obtained from five samples representing different durum varieties grown at Langdon, North Dakota, in 1934, but less satisfactory results were secured from four samples from the 1936 crop. There is no direct comparison between the color characteristics of micro macaroni discs and tubular macaroni in the paper by Cunningham and Anderson (1943a).

The study reported in this paper was conducted to obtain further information regarding the effect of degree and duration of pressure on visual color score of macaroni discs, and to compare directly the color scores of disc and tubular macaroni processed from the same series of semolinas.

Materials and Methods

Purified semolinas used in this investigation were experimentally milled from six varieties of durum wheat grown at Langdon in 1943. Of these, Mindum, Carleton, Stewart, Kubanka, and Monad are grown commercially, while L.D. 153 is still in the experimental stage. These varieties were selected because they represent a wide range in macaroni color.

The wheats were milled in an Allis-Chalmers experimental mill and purified in a small scale purifier, and the tube macaroni was processed from the purified semolina using the methods and technique outlined by Harris and Sibbitt (1942). Micro macaroni discs were also made from the purified semolinas at three pressing times and four pressures as discussed by Smith *et al.* (1946). Since these workers also described details of processing the discs, and making light transmission readings, experimental technique will not be outlined in the present paper. The macaroni and the micro macaroni discs were judged visually against a color standard. The color scale ranged from 0 to 10 in increments of 0.5.

Results

Table I shows the individual visual color scores and the means of the micro macaroni discs for the six varieties at three pressing times and four pressures; also the visual color scores for the tubular macaroni. In processing the discs, 2,000 pounds pressure per square inch for 60 seconds produced discs of satisfactory colors with a fairly good range between the varieties, and was selected as the standard for comparison with the macaroni. This choice is also substantiated by the light transmission readings for the discs at the various times and pressures, presented graphically in the paper by Smith *et al.* (1946). This method has the advantage of being comparatively rapid, and the pressure is very convenient to employ.

An extreme variation in color score is evident among the different treatments, and emphasizes the importance of time and degree of pressure in processing disc macaroni. The poor color score of the discs produced at relatively low pressures illustrates the observation of macaroni manufacturers that the first portions of macaroni extruded from the press, before the optimum pressure for the development of satisfactory color is built up, are dull and have poor color. Smith and associates found that the first sections of tube macaroni from an experimental press contained numerous small air bubbles, and attributed the unsatisfactory color characteristics of the product to this fact. Numerous small bubbles interfere more with the passage of light through the material than a few large bubbles.

TABLE I

VISUAL COLOR SCORE OF MACARONI DISCS
(Durum wheats grown in 1943)

Time	Pressure	Carleton	Mindum	Stewart	L.D. 153	Kubanka[1] 314	Monad[1]	Mean
Sec.	*lb./sq. in.*							
20	1,250	3.0	3.0	3.0	3.0	2.0 D	1.5 DB	2.6
20	1,500	4.0	4.0	4.0	4.0	3.0 D	2.0 DB	3.5
20	2,000	6.0	6.0	6.0	6.0	6.0 D	4.0 DB	5.7
20	3,000	9.0	8.0	8.0	8.0	6.5 D	5.0 DB	**7.4**
Mean								4.8
60	1,250	4.0	4.0	5.0	5.0	3.0 D	2.5 DB	3.9
60	1,500	7.0	7.0	6.0	6.0	6.0 D	5.5 DB	6.3
60	**2,000**	**9.0**	**9.0**	**9.0**	**8.0**	**7.0 D**	**6.0 DB**	8.0
60	3,000	9.0	9.0	9.0	9.0	7.0 D	6.0 DB	**8.2**
Mean								6.6
240	1,250	8.5	8.0	7.0	7.0	6.5 D	5.0 DB	7.0
240	1,500	9.0	9.0	8.0	8.0	7.0 D	6.0 DB	7.8
240	2,000	9.0	9.0	8.5	9.0	7.0 D	6.0 DB	8.1
240	3,000	9.0	9.0	9.0	9.0	7.5 D	6.0 DB	**8.2**
Mean		7.2	7.1	6.9	6.8	5.7	4.6	7.8
Color score of tube macaroni		9.5	7.5	8.5	9.5	7.0	3.0	

ANALYSIS OF VARIANCE

Source of variation	Degrees of freedom	Variance
Between varieties	5	12.39[2]
Between pressures	3	42.05[2]
Between times	2	54.68[2]
Interactions:		
(Varieties × pressures)	15	0.22
(Varieties × times)	10	0.21
(Pressures × times)	6	5.84[2]
(Varieties × pressures × times)	30	0.21
Total	71	

[1] D = dull, DB = dull brown.
Note: Bold type indicates data selected for comparison with tube macaroni.
[2] Denotes significance at 1% point.

An analysis of variance for the visual color score of the micro macaroni discs at the various pressing times and pressures is given in Table I. Very significant differences are shown for varieties, pressures, and times. Varieties have the least effect, with times the greatest. The data are shown graphically in Figure 1. Pressing for 20 seconds apparently cannot develop the best color irrespective of the pressure applied. Pressing for 60 seconds, however, will yield optimum color provided the pressure is above a minimum, approximately 2,000 pounds per square inch in the present instance. A pressing time

of 240 seconds attains maximum color score slightly above 1,500 pounds. The relation between pressure and color score is curvilinear at low pressures for all press times. These results agree rather well with the conclusions of Smith *et al.* (1946), who found similar trends in the effect of duration and intensity of pressure on bubble size and number, and light transmission. The pressure effects on color score are not as sharply defined as they are on bubble properties and light transmission determinations, and this result would be anticipated *a priori* because the latter measurements are quantitative and discrete in character. A correlation coefficient of $+0.743(N=78)$ was found between the light transmission readings and the visual color scores for

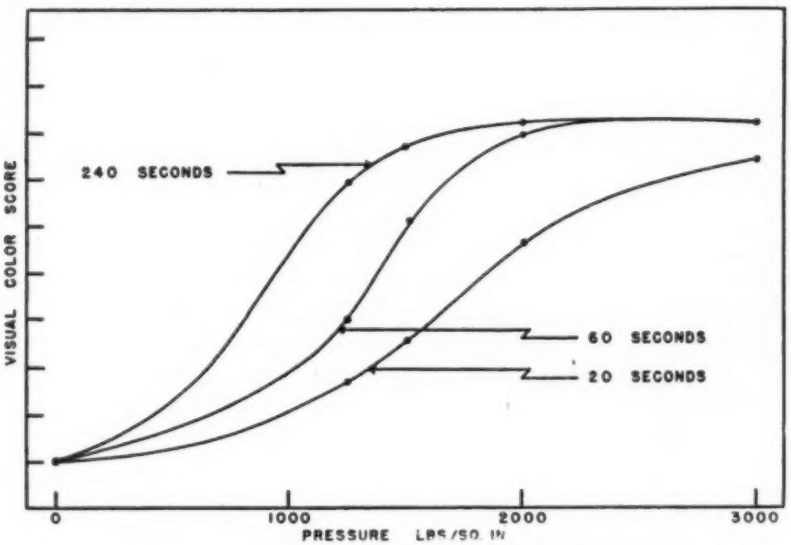


Fig. 1. Effect of pressure and pressing time on the visual color score of macaroni discs. The curves represent the mean values for three varieties.

the micro discs. Although this value is not sufficiently high to allow the accurate prediction of color score of disc macaroni from light transmission readings of the discs, it does show a distinct positive relation between the two series of observations. The lack of better agreement apparently lies in the high pigment content of Monad, which is not reflected in the light transmission values, and in the failure of the visual score to differentiate well among the higher pressures.

Comparative macaroni disc color score data representing six durum varieties are shown in Figure 2. The solid bars represent the mean color scores for macaroni from wheats grown at Langdon for six consecutive years (1940–1945). The finer cross-hatched bars show the results obtained on the tubular macaroni for one year only (1943),

and the coarse cross-hatched bars denote the data for the discs processed from the same semolina using 60 seconds pressing time and 2,000 pounds pressure. These data yield a direct comparison of the color scores of tubular macaroni obtained from six years' data with the results secured from one year's data on the same varieties. As would be expected, some differences in the actual scores are observed, but the general ranking of the varieties is essentially the same. An indication is also given of the relative grading of the two products within one year. In view of the satisfactory relationship in macaroni color between data from six crops and the results from the 1943 crop, and between the tubular and disc macaroni series for 1943, it would appear reasonable to assume that a similar relationship would exist between

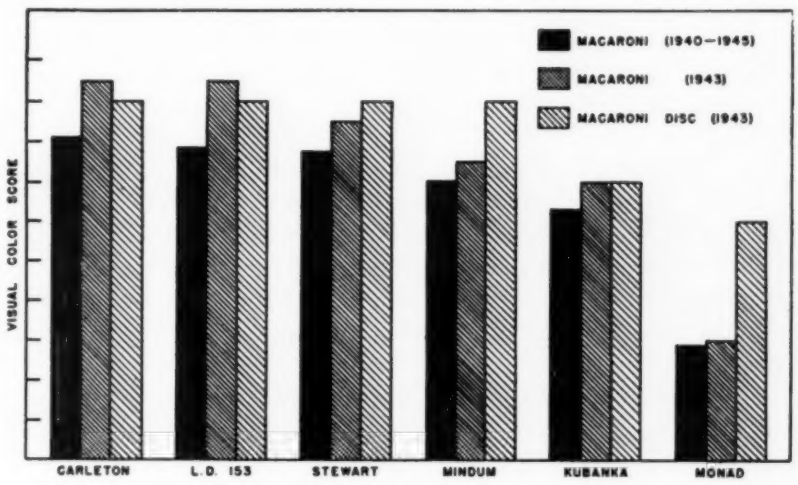


Fig. 2. Visual color scores of tubular macaroni and micro macaroni discs (pressure 2,000 pounds per square inch, pressing time 60 seconds).

tube macaroni and macaroni discs for six years' data. Actually to prove this hypothesis it would be necessary to carry on this study for a number of years. However, with the recent development of a newer and possibly a better method for evaluating small samples of semolina (Martin, Irvine, and Anderson, 1946), the utility of the disc procedure will in all probability be greatly curtailed, especially for nursery samples. It is interesting to note the Mindum and Monad had a higher color score for the micro macaroni disc. In this disc, Monad appeared more translucent than in the tubular macaroni; however, it still possessed the distinct undesirable brown color which is characteristic of this variety. Apparently there is a highly significant positive association between the color scores for the two series of products, but the

number of readings (six) was too small to justify the calculation of a correlation coefficient for subjective data of this nature. It does show, however, that macaroni discs give a good indication of the color ranking of tube macaroni processed from the same material.

Summary

Semolinas were experimentally milled from six durum varieties grown at Langdon in 1943. Tubular macaroni and micro macaroni discs were processed. Three times and four pressures were used in producing the discs.

Very significant differences in the visual color scores of the discs were found between varieties, pressures, and times, with the effect of variety being least marked. A pressing time of 60 seconds and a pressure of 2,000 pounds per square inch gave the most satisfactory results. A satisfactory relationship was evident between the visual color scores of these discs and tubular macaroni made from the same semolina.

A correlation coefficient of +0.743 was found between the light transmission readings and the visual color scores for the micro discs. This value is not high enough to permit the estimation of color score from light transmission readings, owing chiefly to the relatively high pigment content of one of the durum varieties, and to the inability of the visual score to differentiate effectively among the higher pressure treatments.

It is concluded from this study that colors judged from micro macaroni discs could with limitations be used as an index of the color of the actual macaroni.

Acknowledgment

The authors wish to acknowledge the cooperation of Glenn S. Smith in supplying the discs for the visual color examination.

Literature Cited

Anderson, J. A., and Cunningham, R. L.
1943 Micro tests of alimentary pastes. III. The differential response of varieties to processing methods. Can. J. Research **C21**: 265–275.

Cunningham, R. L., and Anderson, J. A.
1943 Micro tests of alimentary pastes. I. Apparatus and method. Cereal Chem. **20**: 171–185.
1943a Micro tests of alimentary pastes. II. Effects of processing conditions on paste properties. Cereal Chem. **20**: 482-506.

Fifield, C. C.
1934 Experimental equipment for the manufacture of alimentary pastes. Cereal Chem. **11**: 330–334.

——, Smith, G. S., and Hayes, J. F.
1937 Quality of durum wheats and a method for testing small samples. Cereal Chem. **14**: 661–673.

Harris, R. H., and Sibbitt, L. D.
1942 Experimental durum milling and processing equipment, with further quality studies on North Dakota durum wheats. Cereal Chem. **19**: 388–402.
Martin, V. G., Irvine, G. N., and Anderson, J. A.
1946 A micro method for making macaroni. **23**: 568–579.
Smith, G. S., Harris, R. H., Jesperson, E., and Sibbitt, L. D.
1946 The effect of pressure on macaroni discs: Size and number of air bubbles in relation to light transmission. **23**: 471–483.

NOTE ON A SEMIMICRO MILLING PROCEDURE FOR DURUM WHEAT [1]

M. H. FISHER and W. O. S. MEREDITH

Grain Research Laboratory, Board of Grain Commissioners for Canada, Winnipeg, Manitoba

(Received for publication August 5, 1946)

The semimicro milling procedure described in this note was developed in the laboratory to provide sufficient semolina for quality tests on early generations of durum wheats produced by plant breeders. Its particular advantages, as compared, for instance, with the methods of Fifield, Smith, and Hayes (1937) and of Harris and Sibbitt (1942), are that it can be used with as little as 300 g of wheat and produces a higher yield of semolina. There is a slight sacrifice in coarseness of the semolina, but this is about the same in granulation as, and equal in dress to, a commercial third sizing, and has proved entirely satisfactory for all laboratory tests of macaroni-making quality. Although the flow was designed primarily for small samples, it is now used in this laboratory for large samples as well.

A minimum of 300 g of clean wheat (14% moisture), plus an additional 1% for scouring loss, is recommended for the semimicro procedure; less wheat produces too small an amount of semolina for efficient purification. The moisture content of the wheat is raised to 14.5% with distilled water and, after mixing thoroughly, the wheat is set aside in a closed container to temper for 18 hours. It is then scoured and the weight reduced to 302 g, which corresponds to 300 g at 14% moisture. Just before milling, additional water is added to bring the moisture content of the wheat to 17%.

The flow is shown in Figure 1, and requires four breaks, two reductions, and four purifications. It was designed for an Allis-Chalmers mill with first and second break rolls mounted two-high in the same housing. The wheat thus passes from the first to the second break

[1] Paper No. 84 of the Grain Research Laboratory and No. 248 of the Associate Committee on Grain Research (Canada).

without intermediate sifting. For a mill with separate stands for first and second breaks, the sifter may be by-passed after the first break which releases very little semolina. The flow may be modified by changing the first two breaks to suit the available stands of break rolls.

The recommended corrugations and spacing of the rolls are as follows: first break, 18 corrugations, .056 inch; second break, 20 corru-

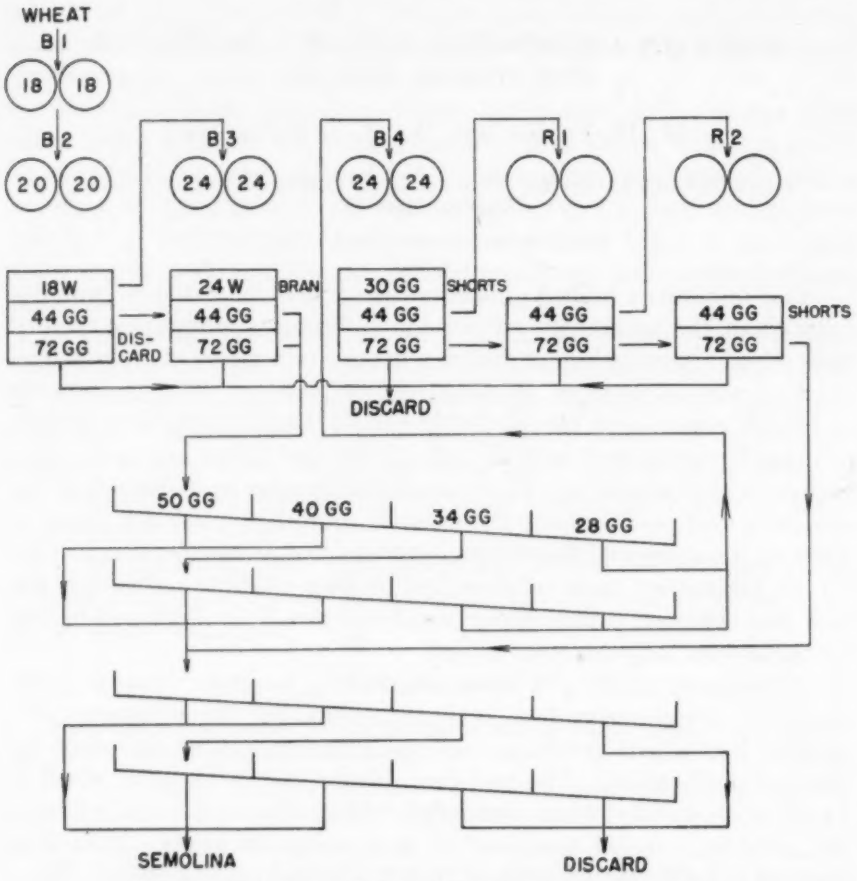


Fig. 1. Flow sheet for milling of wheat and purification of semolina.

gations, .020 inch; third break, 24 corrugations, .008 inch; fourth break, 24 corrugations, .004 inch; first and second reduction, lightly frosted rolls barely touching. All purifications are made with maximum air flow in the machine described by Binnington and Geddes (1936). Other details of the flow are shown in Figure 1.

To obtain information on the comparative yields and quality of semimicro- and macro-milled semolina, 42 varieties of amber durum

wheat were milled by both procedures. Each wheat was a composite sample of the variety grown at several stations in western Canada in different years: 13 varieties in 1941, 16 varieties in 1942, and 13 varieties in 1943. The methods of milling were identical except that 300 g wheat was used for the semimicro procedure and 2,500 g wheat for the macro procedure. Quality was assessed by the disc procedure, described by Cunningham and Anderson (1943), with a dough mixing time of 40 seconds and absorption of 30%. The mean data, for each year and all years combined, for semolina and disc properties are reported in Table I.

TABLE I

MEAN DATA FOR SEMOLINA AND DISC PROPERTIES

Year	Method	Semolina			Discs		
		Yield	Protein	Pigment	Opacity	Yellow[1]	Pigment
		%	%	*p.p.m.*		%	*p.p.m.*
1941	Semimicro	51.6	—	4.16	2.67	48.0	3.24
	Macro	51.5	—	4.19	2.72	49.1	3.25
1942	Semimicro	54.2	12.1	3.25	3.71	41.5	2.54
	Macro	56.1	12.1	3.36	3.41	45.8	2.60
1943	Semimicro	52.2	13.1	5.15	2.72	53.8	3.43
	Macro	52.2	13.1	4.86	2.50	52.9	3.69
1941–1943	Semimicro	52.7	12.6	4.19	3.03	47.8	3.07
	Macro	53.3	12.6	4.14	2.88	49.3	3.18

[1] Reflected light.

The mean values for the 42 samples (last two lines in table) show that semolina yields and pigment contents were essentially the same for both procedures and that protein contents were identical. For the discs, opacity and pigment contents were much the same, but percent yellow was slightly higher for the macro milling. Agreement between the mean values for different years was good for 1941 and 1943 and not quite so good for 1942, particularly in semolina yield and percent yellow. The differences are not unduly large and are considered satisfactory. Because of low protein content, durum wheat of the 1942 crop offered more difficulties in milling and in paste preparation than usual, and the divergence between the mean values found for 1942 was not unexpected.

Although all the factors listed in the table are considered when selecting promising varieties, the visual rating of the discs for color is the most useful single index used. Comparison of varieties is always made with Mindum, the recognized standard variety for amber durum

wheat, and only varieties that are "equal in quality" to Mindum are considered for licensing and distribution. In all three sets of samples, Mindum ranked high in visual appearance of the discs, and there was good agreement between the semimicro- and macro-milled samples on the varieties that were classed as "equal to or better than" Mindum. Some discrepancies were found among the placings of varieties classed as "inferior to" Mindum but, as such samples are generally not recommended for further testing, their actual rank-order placing is of secondary importance.

This study indicates that the semimicro milling procedure is a most useful one; it meets the requirements of the plant breeder and enables the laboratory to provide information on durum wheat quality at a much earlier stage in the breeding program than would be possible otherwise.

Literature Cited

Binnington, D. S., and Geddes, W. F.
1936 Experimental durum milling and macaroni making technique. Cereal Chem. **13**: 497–521.
Cunningham, R. L., and Anderson, J. A.
1943 Micro tests of alimentary pastes. I. Apparatus and method. Cereal Chem. **20**: 171–185.
Fifield, C. C., Smith, G. S., and Hayes, J. F.
1937 Quality in durum wheats and a method for testing small samples. Cereal Chem. **14**: 661–673.
Harris, R. H., and Sibbitt, L. D.
1942 Experimental durum milling and processing equipment, with further quality studies on North Dakota wheats. Cereal Chem. **19**: 388–402.

A MICRO METHOD FOR MAKING MACARONI [1]

V. G. Martin, G. N. Irvine, and J. Ansel Anderson

Grain Research Laboratory, Board of Grain Commissioners for Canada, Winnipeg, Manitoba

(Presented at the Annual Meeting, May 1946; received for publication July 25, 1946)

The first micro method for evaluating the macaroni-making qualities of durum wheats was reported in 1937 by Fifield, Smith, and Hayes, who prepared discs from 30 g of semolina. Since that time, Cunningham and Anderson (1943) have modified the method somewhat and have introduced an optical opacity measurement as a supplementary means of evaluating quality. Further work with a modified form of the apparatus used by Fifield *et al.* has been recently reported by Smith, Harris, Jesperson, and Sibbitt (1946). While the disc

[1] Paper No. 85 of the Grain Research Laboratory, Board of Grain Commissioners for Canada, Winnipeg, and Paper No. 246 of the Associate Committee on Grain Research (Canada).

method has been of considerable value in working with small samples of durum wheats, it has been shown by previous investigators that the correlation betwen the color scores for discs and for macaroni made from the same semolina are not entirely satisfactory. To eliminate the difficulties of working with the disc method while retaining its micro proportions, equipment and method have been developed for producing macaroni from 50 g of semolina. Some of the apparatus used is the same as used in the disc method, some has been modified, and some has been newly designed. In developing the apparatus and method, an attempt has been made to adhere as closely as possible to the routine of commercial practice.

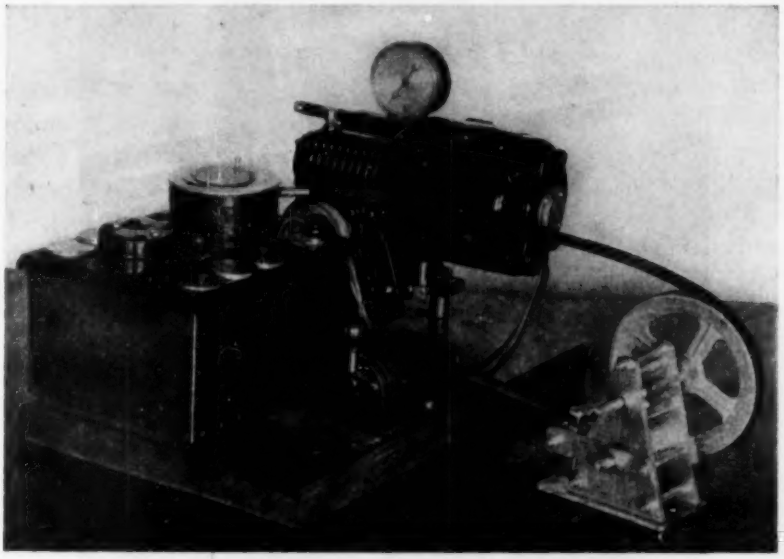


Fig. 1. Mixing unit and belt-driven kneader.

Apparatus

The apparatus can be conveniently described in relation to the five stages of macaroni-making: mixing, kneading, resting in the press, extrusion, and drying.

Mixing Unit. Figure 1 shows the mixing unit. This is essentially the same as that described by Cunningham and Anderson (1943) except for a new mixing spindle and the addition of a vacuum cover over the mixer. The vacuum cover and vacuum gauge are of no concern in this paper; they were added to facilitate experimental work on mixing.

The mixer is directly below the round vacuum cover shown in the photograph. It consists of a small U-shaped mixing trough, 2½ inches

square by 2½ inches deep, set in a constant temperature bath, which forms the bulk of the mixing unit shown in the photograph. The trough contains a horizontal spindle with four mixing paddles, each one inch long and set at right angles to each other; two work flush with the ends of the trough and the other two are spaced equally between the ends. The mixing spindle is driven by a reversible motor at 57 r.p.m., and the direction of mixing may be changed by means of the reversing switch. Lids of six brass semolina-conditioning cylinders

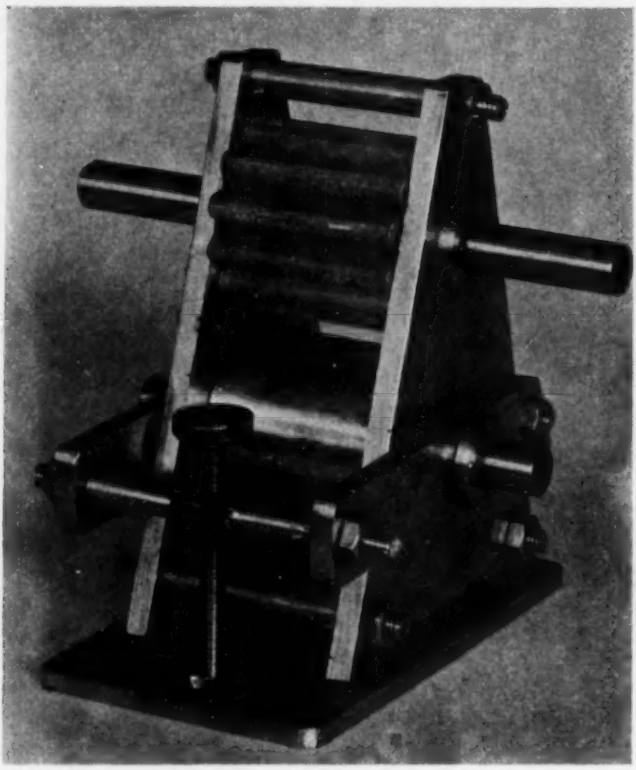


Fig. 2. Belt-driven kneader.

immersed in the bath, the stirrer motor, and the thermoregulator are also shown in the photograph.

Kneader. Figure 2 is a photograph of the kneader. It is made up of two bronze rolls, 2 inches long by 2 inches in diameter, set in a brass framework 5½ inches high. The upper roll is corrugated with 12 blunt teeth ¼ inch deep, and is driven by a belt and pulley from the motor that drives the mixer; the roll rotates at 25 r.p.m. The lower roll is smooth and rotates freely; its distance from the driving roll can

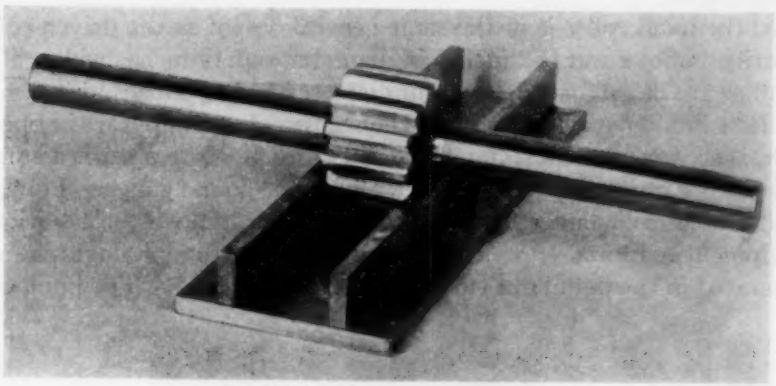


Fig. 3. Hand-operated kneading trough.

be varied by means of the screw adjustment and lever arms in the foreground of the photograph. The space between the rolls can be varied from $\frac{1}{2}$ inch to zero.

Some work has been done with the hand-kneader shown in Figure 3. The trough consists of two brass strips 9 inches long, $\frac{3}{4}$ inch wide, and $\frac{1}{4}$ inch thick, screwed onto a brass plate 9 inches $\times$ 3 inches $\times$ $\frac{1}{4}$

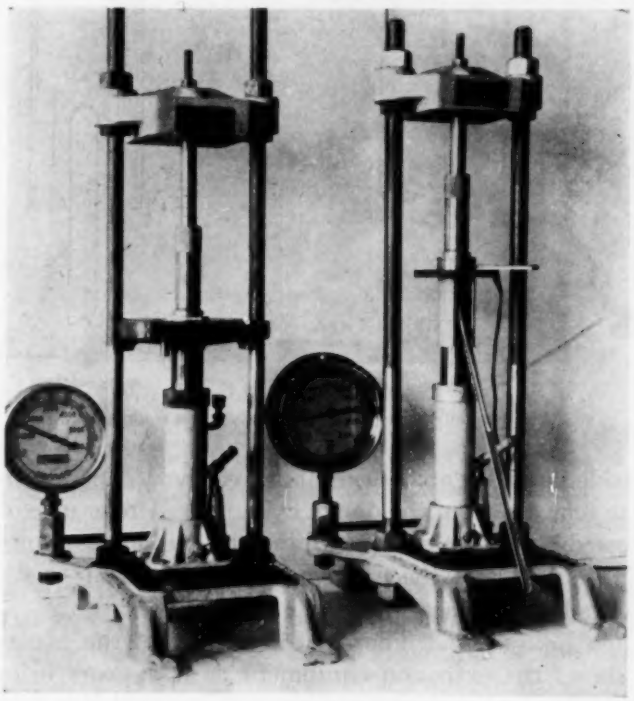


Fig. 4. Rest press, and extrusion press showing macaroni strand emerging from die.

inch; the hand roller is of the same general design as the driven roller described above and is 2 inches in diameter and $1\frac{3}{8}$ inches wide. The handles are of solid steel $\frac{3}{4}$ inch in diameter and $4\frac{3}{4}$ inches long.

Rest Press. A photograph of the rest press, as it appears in operation, is shown on the left-hand side of Figure 4. The press is a standard Carver laboratory press fitted with a 6,000 pound gauge. Plunger, dough holding cylinder, and end cap, which comprise the additional equipment required, are shown assembled in the photograph and separated in the detail drawing at the left in Figure 5. The plunger is

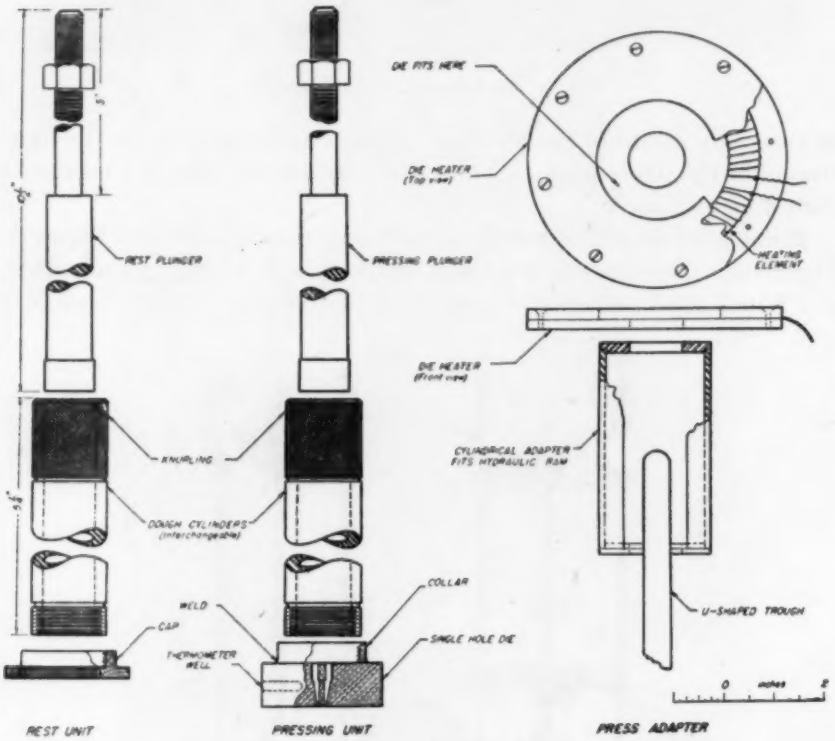


Fig. 5. Detail of rest press and extrusion press equipment.

1 inch outside diameter at the end and is relieved $\frac{5}{8}$ inch from the end to facilitate removal from the dough cylinder. A tolerance of 3/1,000 inch is allowed between the cylinder and the end of the plunger. The cylinder is knurled and the end cap milled to permit easy removal of these parts from the assembly.

Extrusion Equipment. The right-hand side of the photograph in Figure 4 shows the extrusion equipment as it appears in operation. This press is identical with the rest press except that it is fitted with a

2,000 pound gauge. Drawings of the extrusion equipment are shown in detail at the center and right-hand side of Figure 5. The plunger and dough cylinder are identical and interchangeable with those of the rest press apparatus. In addition there are shown the die heater, the single-holed die with threaded collar, and the adapter cylinder fitted with a brass trough. The die was made by F. Maldari and Bros. of New York and is $2\frac{5}{8}$ inches in diameter and $\frac{7}{8}$ inch thick. A bronze threaded collar is welded to the die (with easy-flow silver solder) so

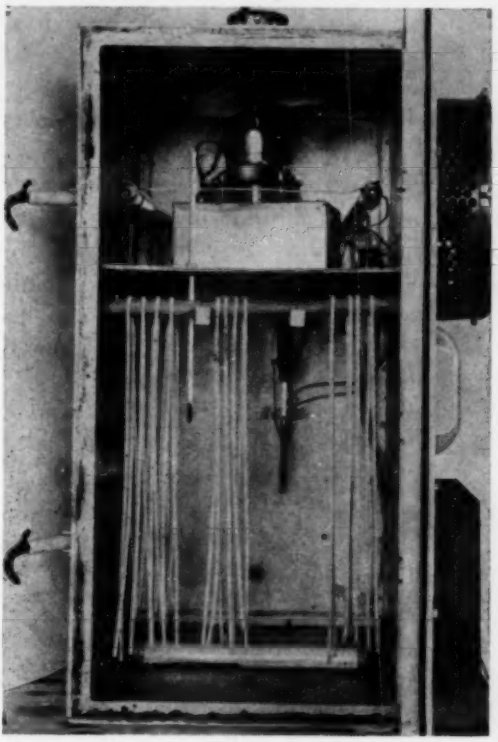


Fig. 6. Experimental small-scale drying cabinet.

that the dough cylinder can be fastened securely to the die. The die heater is made up of a nichrome resistance wire element sandwiched between two discs of tempered masonite, and its temperature is controlled by varying the input voltage by means of a Variac. A trough is necessary to guide the strand of macaroni as it comes from the die and also to prevent it becoming curled and uneven.

Drying Cabinet. Figure 6 is a photograph of the experimental drying cabinet; only a few strands of macaroni are shown in it in order that the controls may be seen. The cabinet is made of tempered

masonite on an angle iron frame, 28 inches high, 21 inches deep, and 14 inches wide. It is divided into two compartments by a shelf slotted with holes to allow free circulation of air from the top to the bottom. The upper compartment contains a small variable-speed fan, a Bahnson humidifier, and two 60 watt 12 inch Lumiline Mazda lamps used as heaters. A third lamp of 40 watts is placed across the bottom of the cabinet. The lower compartment contains racks to hold the wooden drying sticks, an Aminco thermoregulator, and the trough for the wet bulb controller. A junction box is mounted on the side of the cabinet and contains the relays for the humidity controller and the thermoregulator, the rheostat for controlling the fan speed, and switches for all the electrical circuits.

Uniform circulation of air is maintained by the fan. An adjustable inlet vent is provided directly above the fan, and there are two outlet vents, 1 inch in diameter, at the bottom of each of the two sides, about 4 inches from the corners. The humidity is controlled by means of a wet bulb recorder of the type described by Binnington and Geddes (1934). In essence it consists of a recording wet bulb thermometer in which the recording chart is replaced by a metal disc cut to a pattern. When the pen makes an electrical contact with the disc the humidifier is turned on; in this way it is possible to obtain any desired humidity-time gradient.

Method

The method presented below is based on the results of an investigation of the various processing factors; while it produces an excellent sample of macaroni, it is subject to change as and if additional study shows the need.

A 50-g sample of semolina is mixed at 31% absorption (14% moisture basis) for 75 seconds, with the water bath at 86°F. The dough is removed from the mixing trough and run through the kneading rolls 10 times. The rolls are set $\frac{3}{16}$ inch apart, and after each run the dough is folded, alternately crosswise and lengthwise. (If the hand kneader is used, $2\frac{1}{2}$ minutes is sufficient to produce a workable dough.) It is then placed in one of the press cylinders, the bronze end cap is screwed on, and the cylinder is placed in the rest press under a pressure of 1,000 pounds. During the 9 minutes it is allowed to rest, the pressure falls to about 600 pounds. The cylinder is then removed from the rest press and the end cap is unscrewed and replaced by the die. The assembly is then placed in the die heater and mounted on the adapter cylinder. The die is kept warm in the die heater when not in use, and the equilibrium temperature while the dough is being extruded is kept at 105°F. The press is pumped up and the macaroni is extruded at a

constant rate of 36 inches per minute. When the strand of macaroni reaches a length of 30 inches, it is cut and looped over round wooden sticks 4½ inches long and ¾ inch diameter. Four strands are obtained from the dough. These are allowed to case harden in the room for 5 minutes at a temperature of about 75°F and 35% relative humidity, after which they are placed in the drying cabinet maintained at 90°F and 95% R.H. The cabinet conditions remain at this level until one hour after the last sample of a batch is in, after which the humidity controller is set and the relative humidity falls at a uniform rate from 95% to 65% over a period of 48 hours. At the end of this time the heaters and humidifier are turned off and the cabinet is opened. The macaroni is left overnight with the fan running and thus reaches equilibrium with laboratory conditions.

The use of the rest press may be avoided by using an alternative method. The dough is placed in a beaker with a wax paper seal, and this is set for 30 minutes in a desiccator containing water. But this method eliminates the advantages of resting under pressure. The rest press may also be dispensed with by using the extrusion press for both functions, though this more than doubles the time required to process each sample.

Discussion

In working out a suitable method two ideas were kept mainly in mind. Firstly, although the method is a micro one and cannot begin to reproduce the conditions resulting from bulk handling in commercial processing, every attempt has been made to simulate commercial techniques at all stages in the processing. Secondly, the apparatus has been largely built in the laboratory workshop to keep expense at a minimum.

A thorough investigation has been made of all the essential stages of the micro processing to arrive at the method outlined, and some of the more important factors are discussed in the following paragraphs.

Cunningham and Anderson (1943) proposed working with a fixed absorption of 30% in processing discs, and this routine has also been adopted in the macro macaroni technique. It has been found, however, that a higher absorption is necessary for the micro method. With a single strand of macaroni, the effect of the fairly dry atmosphere (35% R.H.) of the laboratory is much more pronounced than when working with many strands. Moreover, when extruded through a heated die, the strand tends to crack readily when processed at 30% owing to the rapid surface drying. The optimum absorption for the micro method appears to be 31%, which produces a strand that is easy to handle and does not stretch if the room humidity is not above 45%.

It should be added, perhaps, that it might be better to work at a room humidity of 65% if this can be maintained, when a slightly lower absorption would be possible.

A fixed absorption is used for the micro test, primarily because material must be conserved. If adequate material is available it is undoubtedly better to work with more than one absorption level. This will show the tolerance of the samples to variations in absorption, and may indicate the level at which individual samples produce the best color.

The decision to rest the dough under pressure was influenced by two factors. The first of these is that this is actually done in the commercial process; the second is that, without this step, maximum efficiency cannot be obtained during the extrusion process. To obtain the best surface on the macaroni, the extrusion pressure should be fairly low. But to obtain a dough that requires comparatively little pressure for extrusion requires either an excessively long rest period, or a shorter rest period combined with pressure on the dough. The rest period was chosen first for convenience, as it fits in with the most efficient timing of the processing; and the optimum pressure for this time was then chosen with due regard to the limitations of the apparatus.

The effect of pressure during resting seems to be twofold: it appears to make the dough more translucent and it decreases its viscosity. The pressure required to extrude the macaroni at a constant rate is a direct function of the viscosity of the dough. Assuming that the die itself is in good condition, the surface of the macaroni depends on the extrusion pressure; the lower the extrusion pressure, the better the surface of the macaroni. Three factors are inversely related to the extrusion pressure: absorption, rest pressure, and die temperature. The values of these factors are all fairly high in the method chosen, and all contribute to a low extrusion pressure and thus improve the surface of the macaroni. The lower limit which can be placed on the extrusion pressure is that at which the dough ceases to weld properly after being forced through the three sections of the die.

It is desirable in testing a new method to compare its results directly with those obtained by commercial processing. It has not yet been possible to compare the macaroni made by the micro method with commercially made macaroni since the commercial method requires so much material that a special series of samples must be grown. Accordingly, it has been necessary to adopt as a standard of comparison the macro method, described by Binnington and Geddes (1936), which has been used in this laboratory. Nine varieties of durum wheats grown in Western Canada in 1945 were available for this study.

Macaroni was processed from these varieties by the two methods. Figure 7 shows a comparison of percentages of yellow and of white in the total reflected light for each of the samples. Agreement between the two methods is considered very good. The reflected red light is not shown as its value is low and is practically the same for all samples; the experimental error in its determination is also quite large, and the differences between samples are not significant.

The micro method is more reproducible than the macro method. This is readily understandable since the degree of control at all stages

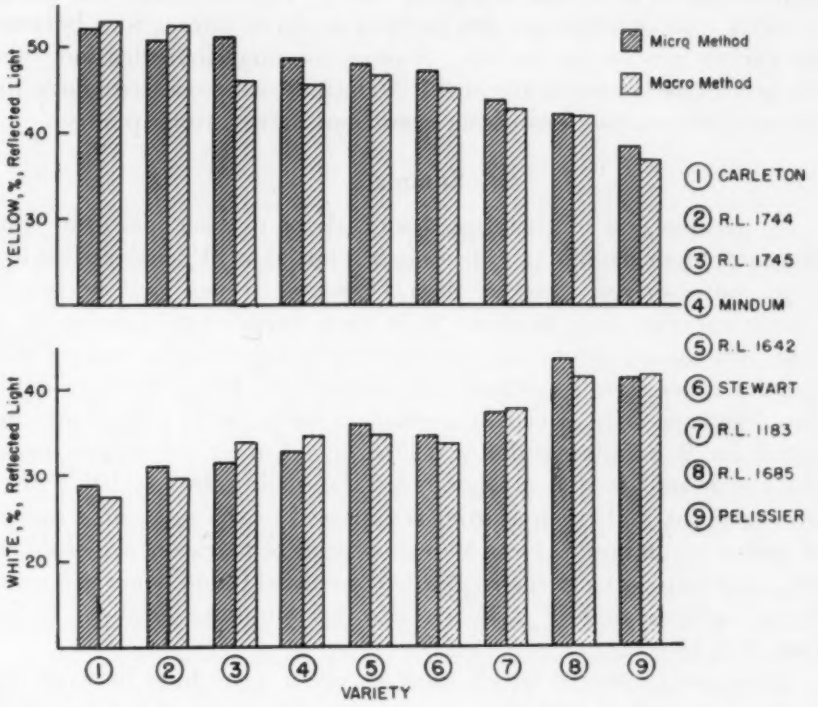


Fig. 7. Comparison of micro and macro methods showing reflected light scores for nine varieties of durum wheat.

is much closer for the former than for the latter. In the micro method it is possible to control and vary all the conditions in the processing: absorption; mixing time and temperature; kneading time and type of kneading; rest time and pressure; extrusion time, pressure, and temperature; die temperature; and rate of drying. All these factors are directly under the control of the operator and all can be held within very close limits. The small size of the drying cabinet also aids in providing more uniform conditions for all samples. A series of 32 samples of macaroni, made from the same semolina and processed in

one batch under identical conditions, had a standard error in pigment content of only ±.09 p.p.m. The surface of the micro macaroni is very similar to that of the commercial product, except that the former contains more air bubbles. This difference has been found with all small-scale tests so far reported and seems to be inherent in the necessity for shorter mixing times and subsequent more intimate and violent working of the dough.

The time required per sample in the micro method is less than half that required by the macro method and thus twice the number of samples can be processed in a given time. This facilitates the use of factorial type experiments and permits study of interactions between the various processing factors. A more thorough investigation, than has previously been possible in the laboratory, can now be made of the effects of the various processing conditions on macaroni quality.

Summary

Apparatus and method are described for making macaroni from 50-g samples of semolina. The dough is mixed at 31% absorption in a small motor-driven mixer with a U-shaped trough and horizontal spindle carrying four blades. It is then kneaded by passing it 10 times through a pair of rolls; the lower roll is free running and smooth, and the upper is motor-driven and corrugated with large blunt teeth. The dough is then placed in a small cylinder, with screw cap, and rested for 9 minutes under pressure in a Carver laboratory press. The cap is replaced by a single-holed die, maintained at 105°F by a heating plate, and the macaroni is extruded in the press at a rate of 36 inches per minute. Four 30-inch strands of macaroni are obtained and, after hardening in the room, they are dried for 48 hours in a small drying cabinet at 90°F with a controlled humidity gradient falling from 95% to 65%.

Data are presented which show excellent agreement between the new micro method and the macro method used in this laboratory. The reproducibility of the micro method is better than that of the macro method, and the quality of the macaroni from the former is equal to that of the latter method.

The apparatus was constructed in the laboratory workshop. It is compact and requires only about 7½ feet of bench space.

Literature Cited

Binnington, D. S., and Geddes, W. F.
1934 Experimental drying equipment for alimentary pastes. Can. J. Research **10**: 221–233.
1936 Experimental durum milling and macaroni making technique. Cereal Chem. **13**: 497–521.

Cunningham, R. L., and Anderson, J. A.
1943 Micro tests of alimentary pastes. I. Apparatus and method. Cereal Chem. **20**: 171–185.
Fifield, C. C., Smith, G. S., and Hayes, J. F.
1937 Quality in durum wheats and a method for testing small samples. Cereal Chem. **14**: 661–673.
Smith, G. S., Harris, R. H., Jesperson, E., and Sibbitt, L. D.
1946 The effect of pressure on macaroni discs: Size and number of air bubbles in relation to light transmission. Cereal Chem. **23**: 471–483.

EFFECT OF GASSING RATE ON LOAF VOLUME [1]

H. Miller, J. Edgar, and A. G. O. Whiteside

Cereal Division, Central Experimental Farm, Ottawa, Canada

(Received for publication May 23, 1946)

The effect of gassing rate in the production of a well-risen loaf has been recognized since the earliest days of cereal science. Although Bailey (1916) put forward the gas production-retention theory, little was published on the subject by other workers for a number of years. Blish and Sandstedt (1927) concluded that fermentation tolerance was associated with the gas production factor. Jørgensen (1931) criticized the A.A.C.C. basic baking formula on the grounds of insufficient gassing power. In the period that followed, numerous workers investigated different aspects of gas production. In reporting their findings, most of the pertinent literature has been cited by such workers as Larmour and Bergsteinsson (1936); Eva, Geddes, and Frisell (1937); Sandstedt and Blish (1939); Eisenberg (1941); and Landis and Frey (1943).

Larmour and Brockington (1934) found that loaf volume is closely related to gas production in the proofing period; studies of gas rate curves and loaf volume showed that various estimations of baking strength would be obtained unless limiting conditions of fermentable sugar during the proofing period are eliminated, particularly with high protein experimentally milled flours.

The present paper deals with the gassing properties of experimentally milled flours of Canadian hard red spring wheat. The studies were undertaken to obtain a more intimate knowledge of the effects of supplementation with sucrose and diastatic malt.

Comparison of Inherent and Supplemented Gassing Rates

The gas production data were obtained with the automatic gas-recording apparatus described by the authors (1943). For the inherent gassing rates of the flour, the formula used was as follows:

[1] Contribution 138 of the Cereal Division, Dominion Experimental Farm, Ottawa, Canada.

	Grams	%
Flour	20.0	100.0
Yeast (bakers' wet compressed)	.6	3.0
Salt	.24	1.2
Water	11.0	55.0

The doughs were fermented for a 6-hour period in the gas-recording apparatus. The "inherent" gassing rate is the average of the three highest successive 10-minute periods of gas production. These peak rates represent the maximum gassing rates attained during the fermentation periods investigated.

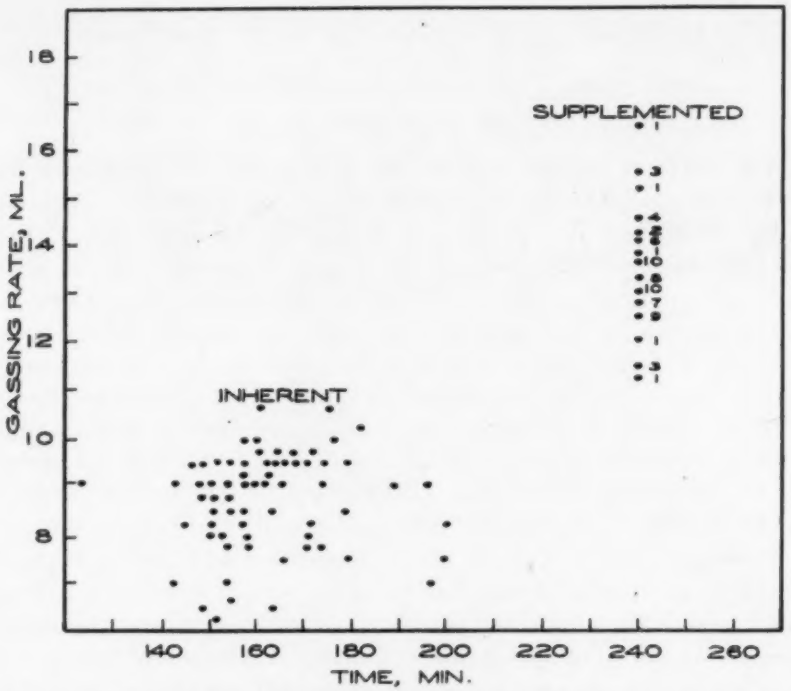


Fig. 1. Maximum "inherent" and "supplemented" gas production of 67 experimentally milled flour doughs. The "inherent" rates represent the mean peak gas production rates and the times at which they occur with doughs containing no added sugar or malt. The "supplemented" rates represent the mean gas production rates during the last 30 minutes of the pan proof period for corresponding doughs supplemented with 3.2% sucrose and 0.24% diastatic malt (250°L). The number of samples reaching the various rates is indicated.

The "supplemented" gassing data were obtained from the gas rates for 20 g flour doughs of the same formula as above plus a uniform supplement of 3.2% sugar and 0.24% diastatic malt (250°L). These "supplemented" doughs were fermented first in the fermentation cabinet according to the A.A.C.C. schedule (*Cereal Laboratory Methods*, 4th ed., 1941) and then placed in the gas-recording apparatus during the last 30 minutes of the pan proof period. The "supplemented"

gassing figure is the mean of the three 10-minute periods of this 30-minute period.

"Inherent" and "supplemented" gassing values were obtained on 67 experimentally milled Canadian hard red spring wheat flours. These values are plotted in Figure 1. The data for "inherent" peak rates show wide variations not only in gassing rates but in the time at which the peak rates were produced. The "supplemented" gassing rates for the doughs at 210–240 minutes or the latter half of the final proof period show that the rates were higher for the "supplemented" doughs than for the "basic" doughs but that the amount of gas produced at oven time is not rendered uniform by the fixed addition of sucrose and malt. Individual increases in gassing rate varied from 1.5 to 8.5 ml. It is interesting to note that over two-thirds of the corrected values lie between 12 and 13.5 ml. However, it is demonstrated that flours vary unequally in their gas response to the fixed supplement and that the "supplemented" rates vary over as wide a range as do the "inherent" rates. Furthermore, it was found that the degree of increase in the "supplemented" rate is independent of the "inherent" rate.

Gas Production and Loaf Volume

The effect of gas production on loaf volume was investigated for a composite experimentally milled straight grade flour from Canadian hard red spring wheat. Four malt-sucrose supplements and five fermentation time schedules were employed. Gassing rates and loaf volumes were determined for each combination in triplicate on three different days. A 700 g flour dough was made up each day using the following formula.

	Grams	%
Flour	700	100
Yeast (bakers' compressed)	21	3
Salt	7	1
$KBrO_3$	.007	0.001
$NH_4H_2PO_4$	.7	0.1
Water	448	64
Malt-sucrose solution	Varied	

The malt-sucrose solution was made up to contain 40 g sucrose and 3 g malt syrup (250°L) in 100 ml. The malt-sucrose solution additions employed were 1, 3, 5, and 8 ml per 100 g flour and corresponded to 0.4% sucrose +0.03% malt, 1.2% sucrose +0.09% malt, 2.0% sucrose +0.15% malt, and 3.2% sucrose +0.24% malt. The five fermentation times employed were 185, 205, 225, 245, and 265 minutes. The doughs were fermented for 105 minutes, punched and divided into five 100 g flour doughs for baking tests and ten 20 g flour doughs for

gas production measurements. The doughs were panned at 130, 150, 170, 190, and 210 minutes, giving a total range in fermentation time of 80 minutes. The punching, panning, and baking schedules are shown below.

Total fermentation	Minutes				
	185	205	225	245	265
First punch	105	105	105	105	105
Second punch	—	125	145	165	185
Panning	130	150	170	190	210
Gassing tests begun	155	175	195	215	235
Into oven	185	205	225	245	265
Out of oven	210	230	250	270	290

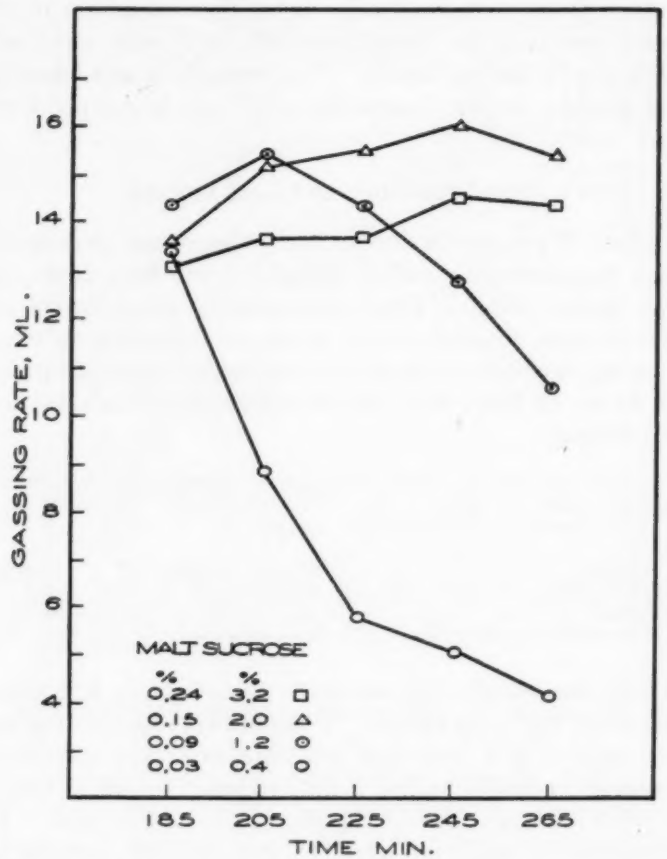


Fig. 2. Response in gassing rates to the addition of different amounts of sucrose and malt (250°L) at varying fermentation times in experimentally milled wheat flour doughs.

In Figure 2 the gassing rates for each malt-sucrose addition are plotted against fermentation time. At 185 minutes all four malt-sucrose supplements gave similar gassing rates ranging between 13 and 15 ml per 10-minute period for 20 g flour doughs. The 1 ml malt-sucrose solution addition showed a marked falling off in gas production as the fermentation time was extended. The 3 ml malt-sucrose solution doughs began to decrease at 245 minutes of fermentation and this was more pronounced at 265 minutes. The doughs containing 5 ml and 8 ml malt-sucrose solution remained high in gassing rate over all periods of fermentation time, but it is interesting to note that the former gave higher gassing rates than the latter at all fermentation times.

The relationship between gassing rate and loaf volume is shown in Figure 3. Low loaf volumes occurred when gas production rates were

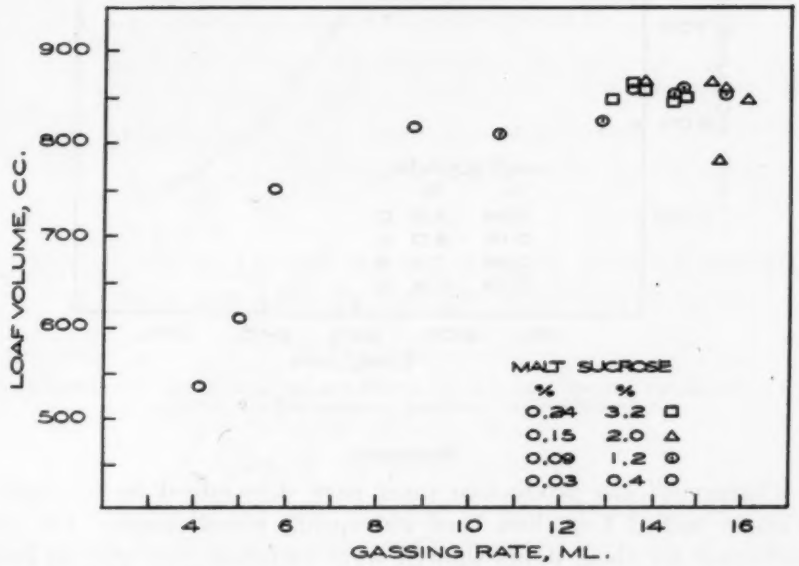


Fig. 3. The influence of gassing rate on loaf volume in experimentally milled wheat flour doughs containing different amounts of sucrose and malt (250°L).

low, but they tended to level out as gas production reached a rate of 12 ml per 10-minute period (20 g flour dough).

Plotting loaf volume against fermentation time, it will be seen in Figure 4 that all malt-sucrose supplementations gave similar loaf volumes at 185 minutes and only the 1 ml malt-sucrose addition showed any marked falling off as fermentation time was extended. The 8 ml malt-sucrose solution maintained loaf volume rather better than the 3 ml and 5 ml supplements over the whole range of fermentation times. Since the loaf volume remained constant at all five fermentation periods

the gassing rate obtained with the 8 ml addition would appear to be the most satisfactory for eliminating gas production as a factor influencing loaf volume. For this particular flour an equally satisfactory result was obtained by using a fermentation period of 185 minutes as with extended fermentation time. The most important factor was the amount of gas being produced in the latter part of the pan proof period.

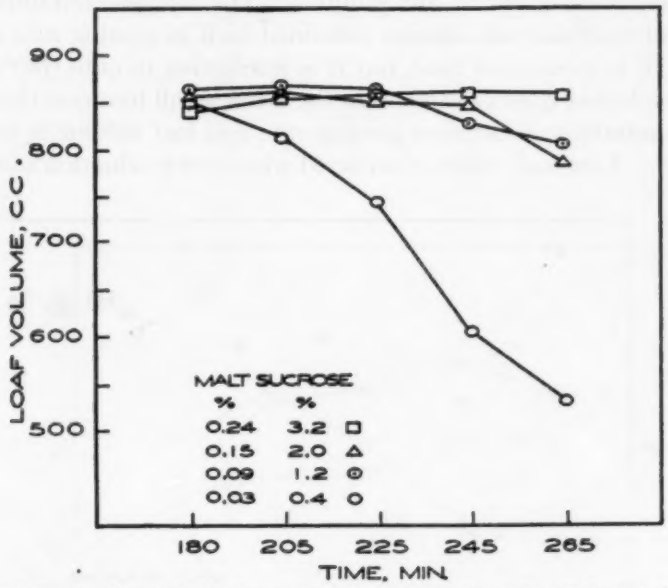


Fig. 4. The influence of fermentation time on loaf volume of experimentally milled wheat flour doughs containing different amounts of sucrose and malt (250°L).

Summary

"Inherent" gas production rates were determined on 67 experimentally milled Canadian hard red spring wheat flours. The gas production for these flours showed wide variations not only in peak rates but in the times at which these peaks occurred. The addition of sucrose and diastatic malt to the doughs increased the gassing rates as fermentation was extended to the pan proof period, but the degree of response was independent of the "inherent" rates.

The relationship of gas production and loaf volume was investigated on a composite experimentally milled flour using four different malt-sucrose supplements and five different fermentation times. Small additions of sucrose and diastatic malt gave high loaf volumes with short fermentation times, but larger additions gave uniform loaf volumes over a fermentation period extending from 185 minutes to 265 minutes. The maintenance of adequate gas production in the pan proof period resulted in high loaf volumes.

Literature Cited

Bailey, C. H.
1916 A method for the determination of the strength and baking qualities of wheat flour. Ind. Eng. Chem. **8**: 53–57.
Blish, M. J., and Sandstedt, R. M.
1927 Factors affecting interpretation of experimental baking tests. Cereal Chem. **4**: 291–299.
Eisenberg, S.
1941 Gas production in yeast fermentation and its applications. III. The baking test. Cereal Chem. **18**: 267–299.
Eva, W. J., Geddes, W. F., and Frisell, B.
1937 A comparative study of various methods of measuring flour gassing power. Cereal Chem. **14**: 458–480.
Jørgensen, H.
1931 On the separation of "gassing power" (diastatic activity) from "strength" in baking tests. Cereal Chem. **8**: 361–374.
Landis, Q., and Frey, C. H.
1943 Direct determination of fermentation rates in dough. Cereal Chem. **20**: 368–376.
Larmour, R. K., and Bergsteinsson, H. N.
1936 Studies on experimental baking tests. III. The effect of various salts on gas-production. Cereal Chem. **13**: 410–420.
—— and Brockington, S. F.
1934 Studies on experimental baking tests. I. Effects of variation in baking formulas on gas production and loaf volume. Cereal Chem. **11**: 451–470.
Miller, H., Edgar, J., and Whiteside, A. G. O.
1943 An automatic gas recording apparatus. Cereal Chem. **20**: 355–361.
Sandstedt, R. M., and Blish, M. J.
1939 The question of sugar levels in laboratory baking. Cereal Chem. **16**: 36–42.

ANALYSIS OF DATA FOR A.A.C.C. CHECK SAMPLE SERVICE. II. PROTEIN AND THIAMINE RESULTS, 1944-45 [1]

W. O. S. MEREDITH

Grain Research Laboratory, Board of Grain Commissioners for Canada, Winnipeg, Manitoba

(Received for publication June 27, 1946)

In summarizing the data obtained in the National Check Sample Service during 1944–45, similar methods were used to those reported by Meredith (1945) and the reader is referred to that paper for details.

Protein

A list of the samples submitted for analysis, together with the mean protein contents, follows:

Sample	Mean protein, %
1. Soft wheat flour	8.18
2. Fine ground whole wheat flour	14.42
3. Low protein family flour	10.67
4. Hard wheat enriched flour	10.70
5. Low grade Red Dog flour	13.06
6. Soft wheat cake flour, enriched	7.02
Mean	10.69

[1] Published as paper No. 87 of the Grain Research Laboratory.

Differences between Laboratory Means over Six Samples. In order that collaborators may know how the means for their own laboratories compare with the general mean over all laboratories, which is 10.69%, differences between means for each laboratory and the general mean have been listed in Table I. The laboratories have been listed in

TABLE I

DIFFERENCES BETWEEN LABORATORY MEAN FOR PROTEIN CONTENTS OF SIX SAMPLES AND GENERAL MEAN FOR ALL LABORATORIES

(Laboratories are identified by the first number in each column)

Range of difference from general mean												
−.25		−.16 to −.07		−.06 to −.04		−.03 to +.02		+.04 to +.06		+.07 to +.15		+.25 to +.37	
Lab. No.	%	*Lab. No.*	%	*Lab. No.*	%	*Lab. No.*	%	*Lab. No.*	%	*Lab. No.*	%	*Lab. No.*	%
129	−.25	79	−.16	20	−.06	83	−.03	27	+.04	87	+.07	18	+.25
		110	−.15	63	−.06	100	−.03	12	+.04	51	+.07	73	+.26
		13	−.13	94	−.05	42	−.03	70	+.05	68	+.09	97	+.37
		85	−.13	127	−.05	74	−.03	82	+.06	72	+.09		
		104	−.13	17	−.05	33	−.03	16	+.06	28	+.10		
		123	−.09	19	−.05	39	−.02			64	+.11		
		125	−.09	32	−.04	105	−.02			89	+.11		
		35	−.08	75	−.04	107	−.02			56	+.12		
		49	−.08	6	−.04	22	−.02			78	+.14		
		86	−.08	76	−.04	43	−.02			93	+.15		
		24	−.08	120	−.04	92	−.02						
		103	−.07			3	−.01						
		1	−.07			47	−.01						
		13	−.07			108	−.01						
		29	−.07			124	−.01						
		53	−.07			58	0.00						
						5	0.00						
						54	0.00						
						118	0.00						
						128	0.00						
						45	0.00						
						98	0.00						
						48	+.01						
						81	+.02						

order of decreasing negative differences by columns to the lower part of column 4, where the difference is zero, and thereafter in order of increasing positive difference.

The 24 laboratories in the center column are those having means within ±0.03 of the general mean, which is beyond criticism. In the adjacent columns (3 and 5) are the additional 16 laboratories with means within ±0.06% of the general mean, which is good.

The remaining columns require further consideration. The first and last columns contain four laboratories with means differing by ±0.16% or more from the general mean. These are classed as unsatisfactory because statistical analyses show that a difference of ±0.16% from the general mean is significant at the 5% level. Subse-

quent examination of the data showed that the standard error of a single sample (0.19%) was abnormally high and that the "normal" necessary difference was 0.07%. On this latter basis, the 26 laboratories in the second and second-last columns, which report differences from ±0.07 to ±0.16%, must be classified as questionable.

Accordingly, of the 70 laboratories, 40 reported mean values over the six samples that are satisfactory, 26 reported results that are of questionable accuracy, and the remaining four reported results that are classified as definitely erroneous. The corresponding figures for the 1943–44 Check Test Series were 38 satisfactory, 18 questionable, and 12 definitely erroneous, out of 68 laboratories.

Differences between Laboratory Means for Single Samples. An examination of the laboratory means for each individual sample showed standard errors for the six samples of 0.13, 0.27, 0.28, 0.10, 0.20, and 0.14%. Detailed examination showed that the distributions of values for all samples except No. 4 were abnormal, and, after eliminating the 11 values causing the abnormality, the adjusted standard deviations for the samples dropped to 0.09, 0.15, 0.11, 0.10, 0.11, and 0.12%. The pooled standard error of a single determination for the six samples then becomes 0.11%. The corresponding figure obtained last year for five samples was 0.10%.

TABLE II

STANDARD ERROR OF EACH LABORATORY OF DIFFERENCE BETWEEN LABORATORY MEAN AND GENERAL MEAN

(Laboratories are identified by first number in each column)

Range of standard errors of differences							
0.82 to 0.13		0.12 to 0.10		0.09 to 0.07		0.06 to 0.01	
Lab. No.	%	*Lab. No.*	%	*Lab. No.*	%	*Lab. No.*	%
97	.82	51	.12	123	.09	125	.06
110	.62	39	.12	86	.09	3	.06
78	.53	76	.12	107	.09	127	.06
73	.52	124	.12	17	.09	22	.06
16	.32	42	.12	43	.09	98	.06
129	.27	48	.12	72	.08	104	.06
35	.27	89	.12	128	.08	19	.05
113	.23	87	.11	94	.08	103	.05
74	.21	33	.11	108	.08	81	.05
28	.18	70	.11	32	.08	58	.05
63	.17	64	.11	6	.07	53	.05
18	.15	92	.11	13	.07	100	.05
24	.15	79	.10	12	.07	83	.04
85	.15	120	.10	27	.07	29	.04
75	.14	68	.10	45	.07	105	.04
54	.13	56	.10			1	.04
93	.13	5	.10			47	.03
		82	.10			49	.01
		20	.10				
		118	.10				

Variability within Laboratories. Differences between laboratory means over six samples do not give an account of the intralaboratory errors. Thus two laboratories might have mean differences of zero, yet one might have had +0.1 for three samples and −0.1 for the other three, while the other had differences of 0.0 for all six samples. The consistency of the laboratories can be compared by listing the standard errors of the mean difference from the general mean for each laboratory, and these are given in decreasing order in Table II. The mean standard error within laboratories was 0.19% and the range was from 0.01 to 0.82%. Statistical analyses showed that there were significant differences between the standard errors and that the 17 values listed in the first column of Table II can be considered abnormal. When these are removed, the mean standard error of the remainder is 0.09%, which is identical with the value obtained last year.

Discussion. Comparison of the results obtained this year with those obtained last year suggests that there has been some slight improvement in the data for protein content.

Thiamine

Only 19 laboratories reported thiamine results for all six samples. The differences between the general mean and the laboratory mean for each laboratory are given in Table III. Last year it was found that the mean values for the laboratories ranged from 2.39 to 3.74 mg/lb,

TABLE III

COMPARISONS AMONG LABORATORIES FOR MEAN DIFFERENCE FROM GENERAL MEAN AND STANDARD ERROR OF DIFFERENCE FOR THIAMINE CONTENT IN MG/LB OF SIX SAMPLES OF FLOUR

Lab.	Mean difference from general mean	Standard error of difference	Lab.	Mean difference from general mean	Standard error of difference
	mg/lb	*mg/lb*		*mg/lb*	*mg/lb*
24	−0.25[1]	0.10	100	+0.04	0.08
16	−0.17[1]	0.15	117	+0.06	0.10
94	−0.16[1]	0.13	5	+0.08	0.20
6	−0.11	0.11	123	+0.08	0.39[2]
89	−0.11	0.22	108	+0.09	0.10
63	−0.09	0.15	13	+0.12[1]	0.10
92	−0.06	0.08	51	+0.12[1]	0.09
22	−0.06	0.14	20	+0.24[1]	0.18
125	0.00	0.13	78	+0.24[1]	0.13
113	+0.01	0.21			

General mean over all laboratories and samples..... 2.19 mg/lb
Original mean standard error of single sample..... 0.17 mg/lb
Normal mean standard error of single sample after reduction from heterogeneous series..... 0.14 mg/lb

[1] Significantly different from general mean.
[2] Significantly different from normal standard error.

a difference of 1.35 mg/lb. This year the laboratory means ranged from 1.94 to 2.43, a difference of 0.49 mg/lb, which represents a substantial improvement.

The standard error of a single sample was 0.52 mg/lb last year and 0.17 mg/lb this year. With the removal of the results of the laboratory which showed the greatest variability, the standard error was reduced to 0.14 mg/lb as compared to the corresponding figure of 0.30 mg/lb last year. On the basis of a standard error of 0.14 mg/lb, the necessary difference from the mean is 0.12 mg/lb, and the seven laboratories marked with footnote 1 in Table III may be considered to have given questionable mean values. The variability within laboratories was examined and the standard error of the laboratory's mean difference from the general mean was included in Table III. Only one laboratory (marked with an asterisk) was shown to give an abnormal standard error for the difference. This again compares favorably with last year when seven laboratories gave abnormal standard errors.

TABLE IV

MEAN, STANDARD DEVIATION, AND COEFFICIENT OF VARIABILITY FOR EACH SAMPLE FOR THIAMINE MG/LB

Sample	Mean thiamine	Degrees of freedom	Standard deviation	Coefficient of variability
	mg/lb		*mg/lb*	*%*
1	1.67	18	0.22	13.2
2	2.05	18	0.21	10.2
3	2.07	18	0.15	7.2
4	2.03	18	0.15	7.4
5	3.07	18	0.24	7.8
6	2.22	18	0.22	9.9
Mean	2.19		0.20	

The standard deviations within samples are shown in Table IV; these were found to be normal. In general, the coefficients of variability for the six samples show considerably greater uniformity than was attained with the six samples studied in 1943–44. This also indicates improvement in the determination as a whole.

Discussion. The improvement achieved during the past year in the thiamine determination should be extremely encouraging to all those who have worked on this determination. Its precision is not yet of the order of that achieved in such standard determinations as that of protein, but there is every reason to believe that a further improvement will be shown next year, and in time the precision of the thiamine determination may well rank with that of other standard methods.

Summary

Seventy laboratories analyzed all six of the 1944–45 A.A.C.C. Check Samples for protein content, and differences between laboratory means were demonstrated. The original standard error of a single sample was 0.19% protein, and 94% of the laboratories agreed within ±0.16% of the general mean value of 10.69% protein for the six samples. The standard error was found to be abnormal and the normal standard error was 0.09% protein; 57% of the laboratories agreed within ±0.07% of the mean protein value. The variability within laboratories was also examined and 17 laboratories showed abnormally high variability.

The data for the thiamine determination, which represent results from 19 laboratories, also showed differences between laboratory means. The original standard error of a single sample was 0.17 mg/lb thiamine and 72% of the laboratories agreed within 0.14 mg/lb of the general mean value of 2.19 mg/lb thiamine for the six samples. This standard error was abnormal and was reduced to 0.14 mg/lb by removal of the result for one laboratory that showed high variability; 63% of the laboratories agreed within 0.12 mg/lb of the mean thiamine value.

The protein results show a slight improvement over those obtained in the previous year, and the thiamine results show a substantial improvement over those for the previous year both in agreement between laboratories and in intralaboratory variability.

Acknowledgment

The interest of Dr. J. Ansel Anderson, Chief Chemist of this laboratory, in the study and his suggestions during preparation of the manuscript are gratefully acknowledged.

Literature Cited

Meredith, W. O. S.
1945 Analysis of data for A. A. C. C. check sample service. I. Protein and thiamine results, 1943–44. Cereal Chem. 22: 437–448.

MODIFICATION OF THE GAS PRODUCTION AND GAS RETENTION PROPERTIES OF DOUGH BY SOME SURFACE-ACTIVE, REDUCING, AND OXIDIZING AGENTS [1]

EMERY C. SWANSON [2] and C. O. SWANSON

Kansas State College, Manhattan, Kansas

(Received for publication May 7, 1946)

The modification of wheat flour dough by chemical means is of importance, especially since wheat varieties are raised which are superior in agronomic properties but whose flours make doughs of in-

[1] Contribution No. 131, Department of Milling Industry.
[2] Present address: International Milling Company, Minneapolis 1, Minnesota.

ferior handling properties. Swanson and Andrews (1942, 1944, 1945) studied the influence of surface-active and reducing agents on the behavior of dough in mixing. They found that the surface-active agents increased the optimum mixing time and decreased the effects of overmixing, whereas reducing agents had the opposite effect. Thus, Chiefkan flour was modified by these wetting agents to produce mixogram patterns similar to those obtained from Turkey or Tenmarq flours; and, likewise, reducing agents produced mixogram patterns from Tenmarq flour similar to those from Chiefkan flour.

A review of the literature regarding the effect of oxidizing and reducing agents on wheat flour has been given in a paper by Shen and Geddes (1942). A supplemental review which brings the literature citations up through 1945 was made by Howe (1946).

In addition to their effects on mixing characteristics, the influence of chemicals on gas production and gas retention is of importance in breadmaking. This paper reports experiments on the influence of surface-active, reducing, and oxidizing agents on gas production and gas retention.

Materials and Methods

The recording apparatus described by Working and Swanson (1946), which provides continuous gas production and gas retention data, was used.

A commercial bakers' hard red winter wheat flour (11.9% protein, 0.44% ash, and 11.4% moisture) and a "lean" formula (flour, water, 2.5% yeast) was used throughout. The only variations were in the kind and amount of chemical agent added.

The surface-active agents used were Aerosol OT,[3] Santomerse S,[4] and Tergitol penetrant 08.[5] The reducing agents used were cysteine hydrochloride, glutathione, and sodium sulfide, and the oxidizing agents, potassium iodate, potassium bromate, and hydrogen peroxide. Ten concentrations of each agent were employed, in addition to the controls. The range of concentrations varied in equal intervals from 60 to 600 mg per 100 g of wheat flour for the surface-active agents, from 6 to 60 mg for the reducing agents, and from 3 to 30 mg for the oxidizing agents. These ranges were sufficiently wide to insure uncovering any effect that might result from the addition of these agents, and are similar to the concentrations used by Swanson and Andrews (1942, 1944, 1945) in their mixogram studies.

A uniform mixing time of two minutes was used for each dough. This period was established as optimum for the flour without the

[3] American Cyanamid & Chemical Corporation, New York, N. Y.
[4] Monsanto Chemical Company, St. Louis, Missouri.
[5] Union Carbide and Carbon Corporation, New York, N. Y.

addition of any of the chemical agents investigated. A uniform period would eliminate the influence of mixing time and, consequently, not interfere with the dough development factor except as the mixing requirements were modified by the agent used. A 10-g aliquot was used to determine gas production and another one for the determination of gas retention. The 90 combinations, 9 chemicals and 10 concentrations of each, were made in triplicate, along with 27 controls. The sequence of the combinations and control samples, during the course of the experiment, was by random selection.

The gas production data have been reported as "initial rate of production" (ml/hour/170 g dough[6]) and "duration of the initial rate" (hours) and the gas retention data as the "maximum amount of gas retained" (ml/170 g dough) and the "time to reach the maximum" (hours). The initial rate of gas production is the average rate during which an adequate supply of fermentable sugar is present and is maintained until the supply becomes so small as to be the limiting factor in fermentation. This stage is shown by an abrupt decrease in the slope of the production curve. The *time* elapsing from the start of fermentation to the change in the slope is the "duration of the initial rate." Maximum gas retention occurs when the rate of production is equalled by the rate of escape from the dough. Prior to maximum retention the rate of production exceeds the rate of loss and the *amount* retained is increasing. After the point of maximum retention has been reached the rate of production has been surpassed by the rate of escape and the amount of gas retained is decreasing. Maximum retention is represented by the highest point on the retention curve (Working and Swanson, 1946). The time elapsing from the start of fermentation to this point is the "time to maximum retention."

Results

For convenience in presenting and discussing the four phenomena studied, namely, initial rate of fermentation, duration of initial rate, maximum gas retention, and time to maximum retention, they will usually be referred to as "rate," "duration," "retention," and "time," respectively. The mean data are shown graphically in Figures 1, 2, and 3. The significant correlations for the effects of the amount of the chemical agents on these four factors have been listed in Table I. Of 36 possible correlations, 13 were nonsignificant, 5 were significant, and 18 were highly significant.

All but five of the 23 significant relationships indicated that the effects of the chemical agents were directly proportional to their con-

[6] 170 g being the average weight of an unfermented pup-size dough.

TABLE I

SIGNIFICANT CORRELATION COEFFICIENTS FOR THE EFFECTS OF CHEMICAL AGENTS ON (*a*) INITIAL RATE OF PRODUCTION, (*b*) DURATION OF INITIAL RATE, (*c*) MAXIMUM AMOUNT OF GAS RETAINED, (*d*) TIME TO REACH THE MAXIMUM

Chemical agents	Correlation coefficients			
	Production		Retention	
	Initial rate	Duration of initial rate	Maximum amount	Time to maximum
Aerosol OT	−0.736	—	−0.959	−0.905[1]
Santomerse S	—	−0.862	−0.892	—
Tergitol pen. 08	−0.892	+0.612	—	+0.822
Cysteine hydrochloride	—	—	−0.950[1]	−0.830[1]
Glutathione	−0.843	+0.640[1]	−0.838	−0.853
Sodium sulfide	—	−0.859	−0.791	−0.835
Potassium iodate	—	—	—	—
Potassium bromate	−0.692	+0.707	+0.860[1]	+0.929
Hydrogen peroxide	+0.624	—	−0.908	—

NOTE: 0.602 = r at 5% level
0.735 = r 1% level.
[1] Curvilinear correlation requiring:
0.632 at 5% level
0.765 at 1% level.

centration. Of the five, cysteine produced a maximum effect on time and bromate a maximum effect on retention; Aerosol caused an accelerated decreasing effect on time and cysteine on retention beyond a certain concentration; glutathione caused an accelerated increasing effect on duration.

Figure 1 shows the effects of the surface-active agents, Aerosol, Santomerse, and Tergitol, on gas production (rate and duration), retention, and retention time. Aerosol markedly lowered the rate and, from the graph, seemed to have a decreasing influence on duration which was, however, not substantiated by regression analysis. Retention was lowered but time was not decreased until the concentration exceeded 0.3 g Aerosol per 100 g flour. Santomerse had no influence on production rate or retention time, but markedly decreased duration of the rate and maximum retention. Tergitol decreased the rate, increased duration and time, but was without influence on retention.

The effects of the reducing agents are shown in Figure 2. Cysteine had no effect on gas production but it decreased gas retention. Cysteine exhibited an optimum concentration in affecting retention time, although the increase on retention time at the lower concentrations was not as marked as the decrease at the higher concentrations.

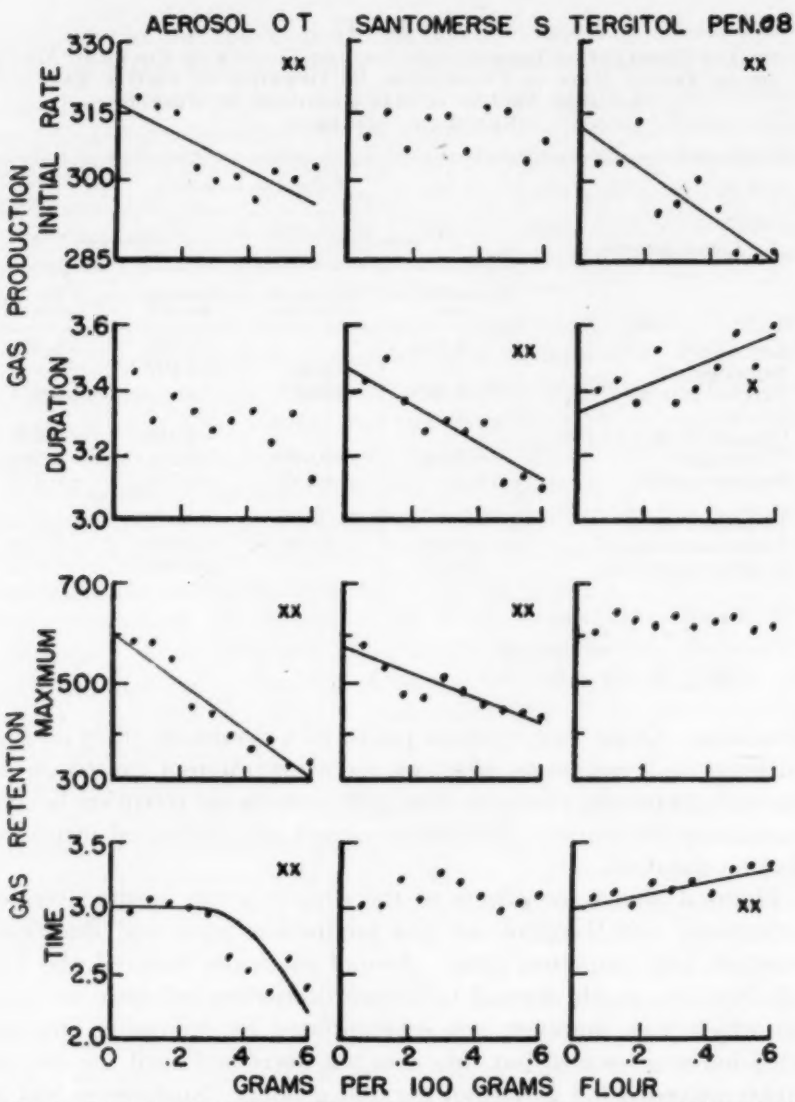


Fig. 1. Effect of surface-active agents on gas production and gas retention. Initial rate is given in milliliters per hour per 170 g dough, maximum retention in milliliters per 170 g dough, and duration of rate and time to maximum retention in hours. Regression lines are shown for all significant relationships.

Glutathione decreased the rate, retention, and time; but increased the duration of gas production rate when the concentration was sufficiently great. Sodium sulfide had no influence on production rate, but decreased the duration. This reagent caused a slight decrease in maximum retention and markedly decreased retention time.

The effects of the oxidizing agents are shown in Figure 3. Potassium iodate was entirely without influence on gas production and

gas retention. Potassium bromate, according to the regression equation, had a depressing action on production rate but the positions of the points indicate some doubt. The duration was increased slightly by bromate and an optimum maximum retention was attained with about 15 mg per 100 g flour; the time to maximum retention was also increased as the dosage of bromate was increased. Hydrogen peroxide apparently increased the rate of production, but had no influence on

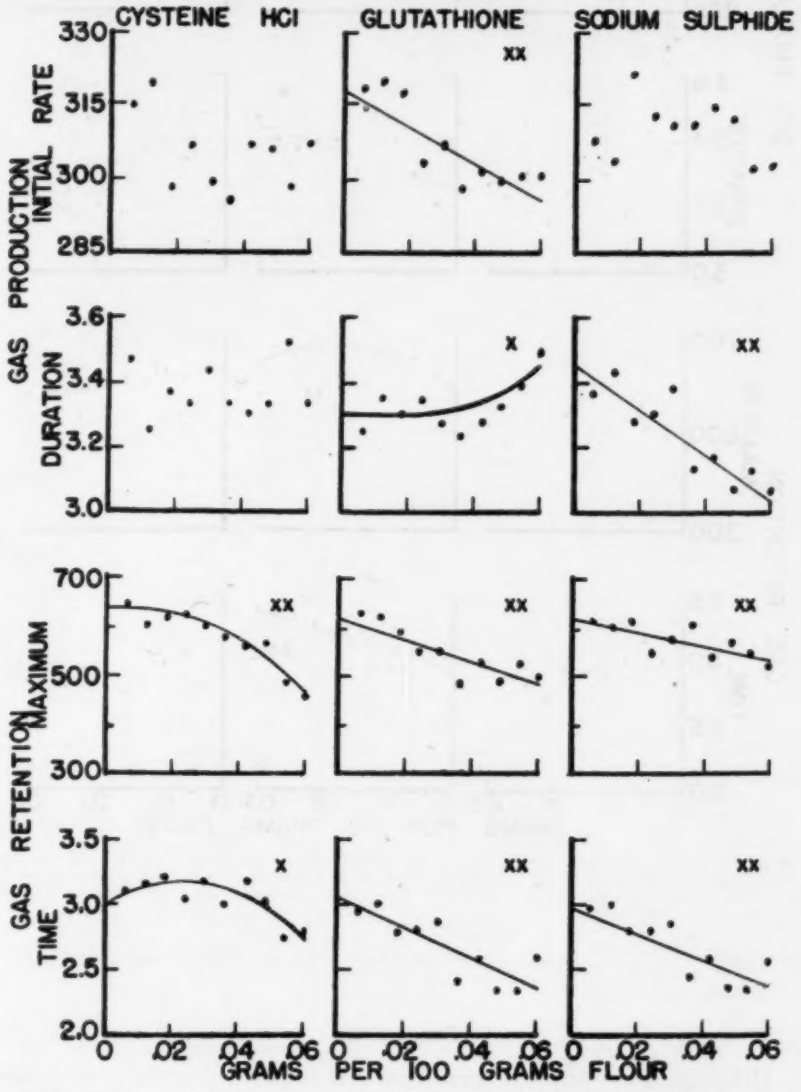


Fig. 2. Effect of reducing agents on gas production and gas retention. Initial rate is given in milliliters per hour per 170 g dough, maximum retention in milliliters per 170 g dough, and duration of rate and time to maximum retention in hours. Regression lines are shown for all significant relationships.

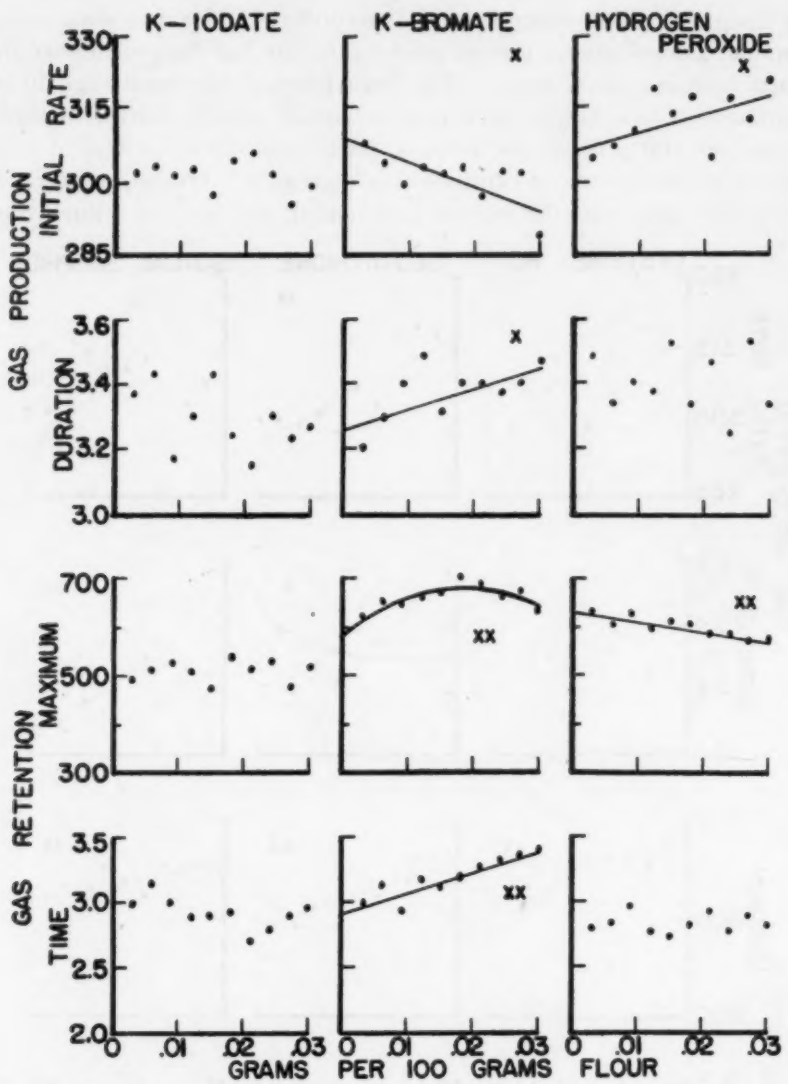


Fig. 3. Effect of oxidizing agents on gas production and gas retention. Initial rate is given in milliliters per hour per 170 g dough, maximum retention in milliliters per 170 g dough, and duration of rate and time to maximum retention in hours. Regression lines are shown for all significant relationships.

duration. It decreased retention but had no effect on the time to maximum retention.

Discussion

Although the results reported in this paper are preliminary, some general observations on the action of the agents used on gas production and gas retention may be made.

Gas Production. Two factors are predominant in gas production, namely, enzymic action and starch susceptibility. The enzymic activities are divisible into two groups, those which hydrolyze starch to sugar and those which ferment sugar. A decrease in the initial rate of gas production would indicate an inhibiting effect on yeast growth and/or the fermentation enzymes inasmuch as adequate sugar was apparently present. An increase in rate, conversely, would indicate an accelerating effect. Of the surface-active agents, Aerosol and Tergitol had an inhibiting effect on rate but Santomerse was without effect. Of the reducing agents, glutathione was the only one which inhibited the rate of production of gas; cysteine and sodium sulfide were inactive. Of the oxidizing agents, the bromate was depressing and the peroxide accelerating, but the iodate was inactive.

A change in the duration of the initial rate of fermentation could result from either, or both, of two actions: an inhibiting action on the starch hydrolyzing enzyme system and/or a change in the starch susceptibility. Aerosol did not have any significant effect on duration, but Santomerse decreased it and Tergitol caused an increase. Of the reducing agents, cysteine had no effect on duration, sodium sulfide shortened it markedly, and beyond the medium concentration used, glutathione increased the duration. Of the oxidizing agents, bromate lengthened the duration, but iodate and peroxide were without effect.

Gas Retention. Gas retention differs from gas production in that an adequate dough structure is requisite for gas retention, but not for gas production. Amount of mixing has a marked influence on such a structure (Swanson and Swanson, 1946), but all doughs were given the same mixing treatment in this study in an attempt to determine the effects of chemical agents without the interference of the mechanical dough-development factor.

Chemicals that influence gas retention are effective, primarily, through their capacity to alter the physical structure of the dough. In addition, the rate of gas production influences gas retention; an increase in the rate of production increases the amount of gas retained owing to the greater rate of escape necessary before the loss balances production. Hence, a chemical which increases or decreases the rate of production would increase or decrease the maximum gas retention.

A part of the decrease in gas retention from Aerosol was due to the decrease in rate of production. However, the decrease in rate cannot account for all the loss in retention. If all the loss in retention were attributable to decreased rate, fermentation would have to continue for about 15 hours, with the maximum retention coming at that time (20 ml/hour rate decrease; 300 ml retention decrease), but no fermentation period was continued for more than 8 hours, and the time to

maximum retention in no case exceeded 3½ hours. This indicates that Aerosol had an appreciable detrimental effect on the dough structure, with respect to the gas-retaining capacity. A similar effect is attributable to Santomerse; the decrease in gas retention was not quite as great although Santomerse was without influence on the rate of production. Therefore, the detrimental effect of Santomerse apparently was about the same as that of Aerosol. The gas-retaining ability of dough was seemingly not affected by Tergitol. However, since a marked decrease in the production rate occurred without any concurrent decrease in gas retention, it is logical to assume that were the production rate maintained constant, Tergitol would show an increase in maximum gas retention.

Most of the detrimental effect of glutathione on gas retention can be accounted for by its depressing action upon the rate of gas production. Since cysteine and sodium sulfide had no influence on gas production rate, their effect on gas retention must be interpreted as due largely to some harmful alteration in dough structure.

Of the oxidizing agents, bromate appears to exhibit an optimum concentration in relation to maximum gas retention. Bromate improved gas retention to a marked extent and when compensation for the seeming reduction in production rate is made, the bromate curve indicating increased retention becomes even more significant. The detrimental effect of hydrogen peroxide on maximum retention was actually more marked than it appears from the graph, since hydrogen peroxide apparently increased the rate of gas production.

Interrelations of Gas Production and Gas Retention. The time to maximum retention is dependent upon both the rate of gas production and the dough structure. Tergitol and potassium bromate had no effect on dough structure, as measured by the gas-retaining capacity of the dough, yet they caused an increase in the time to maximum retention. Aerosol seemed to affect the time factor through both the initial rate of gas production and the retention effects; however, no decrease in time to maximum retention occurred unless the concentration of Aerosol exceeded the medium amount used. On this basis it seems probable that Aerosol would increase retention time up to a certain concentration, if production rate and maximum retention were maintained constant. Santomerse appeared to have no influence on the time to maximum retention in spite of a definite harmful influence upon dough structure, as indicated by a decrease in maximum retention.

With cysteine, an optimum concentration for the time to maximum retention was noted. The decreasing effect of cysteine on time as the concentration was increased beyond the optimum was abetted by its

harmful effect on dough structure, as measured by maximum retention. The decrease in gas retention due to glutathione was apparently caused by both a decrease in the rate of gas production and by an unfavorable effect on dough structure. Sodium sulfide reduced gas retention principally by its harmful action on dough structure.

Of the oxidizing agents, iodate and peroxide had no effect upon retention time, but bromate markedly increased the time, in spite of causing a decrease in rate of gas production. The evidence indicates that bromate has an appreciable influence in the improvement of the dough structure for gas-retaining purposes.

Summary

A commercial bakers' hard red winter wheat flour and a flour-water-yeast formula were used to study the effects of surface-active, reducing, and oxidizing agents on gas production and gas retention. The surface-active agents were Aerosol OT, Santomerse S, and Tergitol penetrant 08; the reducing agents were cysteine, glutathione, and sodium sulfide; and the oxidizing agents were potassium iodate, potassium bromate, and hydrogen peroxide.

These preliminary studies have shown variable effects of the agents used on both gas production and retention. The initial rate of gas production was decreased by Aerosol, Tergitol, glutathione, and apparently bromate; and increased by peroxide. The duration of the initial rate was decreased by Santomerse and sodium sulfide, and increased by Tergitol, bromate, and glutathione (beyond a certain concentration). The maximum gas-retaining capacity of dough was decreased by Aerosol, Santomerse, cysteine, glutathione, sodium sulfide, and hydrogen peroxide, but was increased by bromate (up to a certain concentration). The time elapsing from the start of fermentation until maximum gas retention was reached was decreased by Aerosol (beyond medium concentration), glutathione, and sodium sulfide; and increased by Tergitol and bromate. Cysteine increased the time factor up to medium concentration, but beyond that it caused a decrease.

Literature Cited

Howe, M.
1946 Further studies on the mechanism of the action of oxidation and reduction on flour. Cereal Chem. **23**: 84–88.

Shen, T., and Geddes, W. F.
1942 Protease activity and reducing matter content of wheat flour doughs in relation to baking behavior. Cereal Chem. **19**: 609–631.

Swanson, C. O., and Andrews, A. C.
1942 Factors which influence the physical properties of dough. IV. The effects of surface active agents on the characteristics of the curves made by the recording dough mixer. Cereal Chem. **19**: 102–120.

1944 Factors which influence the physical properties of dough. VI. Effect of cysteine and some other substances on mixogram patterns. Cereal Chem. **21**: 140–149.
1945 Effects of papain, yeast water, cysteine, and glutathione on gluten dispersion or on disintegration as indicated by gluten recovery and by mixogram patterns. Cereal Chem. **22**: 134–149.
Swanson, E. C., and Swanson, C. O.
1946 Gas production and gas retention of dough as affected by type of flour, baking formula, and amount of mixing. Cereal Chem. **23**: 432–440.
Working, E. B., and Swanson, E. C.
1946 An automatic device for the measurement of gas production and gas retention in doughs. Cereal Chem. **23**: 210–216.

DETERMINATION OF UREA AND AMMONIACAL NITROGEN IN FEEDS

MARGARET SCHMOLL and FREDERICK D. SCHMALZ

Russell-Miller Milling Company, Control Laboratory, Minneapolis, Minnesota

(Received for publication March 29, 1946)

Because of the shortage of all types of protein concentrates during the past few years, urea has been used to a greater extent than ever before in dairy and sheep commercial mixed feeds. Upon the recommendation of the Association of American Feed Control Officials, the use of urea and certain ammonium salts has been limited to not more than one-third of the total crude protein nitrogen in feeds for ruminants.

Under the direction of the Associate Referee, W. B. Griem, the Association of Official Agricultural Chemists has published several reports on a method for the determination of urea and nitrogen salts in feeds (Griem, 1941, 1941a, 1942, 1942a, 1944). A recommendation (Griem, 1944) was made that the method as revised (1942) be made official (final action).

In our hands some difficulty was encountered in using this method in routine analyses of urea-containing feeds, particularly those concentrates that had relatively high amounts of urea. Excessive foaming was obtained in the distillation and an unsatisfactory fleeting end point was experienced in the titration. Therefore, certain minor modifications in the method were introduced which make the procedure more reliable. These modifications are essentially the use of sodium hydroxide, a larger amount of calcium chloride, and the introduction of material to prevent foaming and bumping. Glass beads and a small quantity of finely ground mill feed (shorts) served the purpose better than anything else that was tried. The mill feed used for this purpose should, of course, contain no added urea or ammonium salts.

Determination

Reagents:

Calcium chloride: 25% solution
Sodium hydroxide: 10% solution
Urease tablets: 25 mg (a product of Hynson, Wescott, and Dunning, Inc.)
Buffer solution: 41.0 g of sodium acetate and 30.0 g glacial acetic acid per liter
Standard acid: Normality as used in the Kjeldahl nitrogen determination
Standard alkali: Normality same as standard acid
Indicator: Sodium alizarin sulfonate or methyl red
Paraffin
Glass beads
Finely ground mill feed (shorts).

Procedure:

Weigh a 10.0 g sample of the urea-containing feed, place in a 500 ml volumetric flask, and make up to volume. Allow the suspension to settle after shaking and pipette 20 ml of the supernatant liquid into an 800 ml Kjeldahl flask. Add buffer in amounts corresponding to the following levels of urea: 1 to 5% urea—1.2 ml buffer solution; 6 to 10% urea—2.4 ml buffer solution; 11 to 15% urea—3.6 ml buffer solution. Grind two urease tablets in a glass mortar and wash into the flask with not more than 100 ml of water. Agitate gently, taking care not to get the solution up along the sides of the flask. Stopper the flask and allow to digest for two hours at 40°C.

After the digestion add approximately 10 glass beads, a piece of paraffin (0.5 to 1 g) and about 0.5 g of finely ground mill feed to the sample in the digestion flask. Then add 10 ml of 25% calcium chloride solution and 10 ml of 10% sodium hydroxide solution. Add sufficient water to make a total volume not to exceed 350 ml and distill 200 ml into 20 ml of standard acid. Back titrate with standard alkali.

A blank determination should be made using 20 ml of distilled water in place of the 20 ml aliquot of sample. One ml of 0.1 *N* acid is equivalent to 0.0030 g of urea.

Samples of both feeds and solutions containing urea in amounts normally added to feeds were tested. Checks were in close agreement, and the percent of error was comparable to that experienced in regular protein determinations of feeds.

Literature Cited

Griem, W. B.
1941 Changes in the Official and Tentative Methods of Analysis made at the fifty-sixth annual meeting, October 28, 29, and 30, 1940. Outline of Method. J. Assoc. Official Agr. Chem. **24**: 66–101.

1941a Report on ammoniacal and urea nitrogen (feeds). J. Assoc. Official Agr. Chem. **24**: 867–870.
1942 Changes in the Official and Tentative Methods of Analysis—Recommendation of adoption of method as official (first action) with minor changes. J. Assoc. Official Agr. Chem. **25**: 92–93.
1942a Report on ammoniacal and urea nitrogen. J. Assoc. Official Agr. Chem. **25**:874–877.
1944 Report on ammoniacal urea and nitrogen salts. J. Assoc. Official Agr. Chem. **27**: 494–495.

BOOK REVIEWS

Physical Methods of Organic Chemistry. II. Edited by Arnold Weissberger, Eastman Kodak Company. Interscience Publishers, Inc., New York, N. Y. 1946. 631 pp., 15 × 23 cm. Price $8.50.

This second volume of the two-volume series on the subject maintains the excellence of subject matter treatment and of workmanship which has been noted in a review of the first volume (*Cereal Chemistry* **23**: 114, 1946). Pages are numbered consecutively in the two volumes and a subject index (52 pp.) covering both volumes concludes the second volume.

Methods applicable to 10 additional types of physical measurements are treated in this volume. The subject matter, authors, and number of pages devoted to the discussion of each subject are as follows: XVII. Spectroscopy and Spectrophotometry —W. West (86 pp.). XVIII. Colorimetry, Photometric Analysis, and Fluorimetry —W. West (46 pp.). XIX. Polarimetry—W. Heller (120 pp.). XX. Determination of Dipole Moments—C. P. Smyth (22 pp.). XXI. Conductometry—T. Shedlovsky (40 pp.). XXII. Potentiometry—L. Michaelis (64 pp.). XXIII. Polarography—O. H. Muller (98 pp.). XXIV. Determination of Magnetic Susceptibility—L. Michaelis (36 pp.). XXV. Determination of Radioactivity—W. F. Bale and J. F. Bonner, Jr. (42 pp.). XXVI. Mass Spectrometry—D. W. Stewart (24 pp.).

The detailed and critical discussions contained in these volumes of the many types of physical measurements already in common use in the chemical laboratory can lead to a more accurate utilization of these methods. Perhaps an even more important function of these volumes will result from the description found therein of several equally important but as yet little used methods. It is especially appropriate that discussions of methods for isotope estimation should be included in these volumes in view of the probability that so much use will be made of these elements in organic and biological studies in the near future.

D. R. Briggs
Division of Agricultural Biochemistry
University of Minnesota
University Farm, St. Paul, Minnesota

Enzymes and Their Role in Wheat Technology. Edited by J. A. Anderson. American Association of Cereal Chemists Monograph Series. Vol. I. ix + 371 pp. Interscience Publishers, Inc., New York, N. Y. 1946. Price $4.50.

The first volume of this new monograph series incorporates an unusual feature: namely, each enzyme class that is reviewed is presented first from the viewpoint of basic enzymology and secondly, in a twin chapter under a different authorship, from the more specific viewpoint of wheat technology. The chapters dealing with basic enzymology are not limited to cereal enzymes but rather present the broad characteristics of the enzyme classes discussed. The classes of enzymes reviewed are properly limited to those of greatest interest to cereal chemists—amylases, proteases, lipases, oxidases, and the fermentation enzymes.

The chapter on the general chemistry of enzymes by Sandstrom has no companion chapter. This condensed chapter will be of interest primarily to those who have little knowledge of enzymology. It is necessarily incomplete.

The first chapter on amylases by Caldwell and Adams reviews higher plant, animal, *Aspergillus oryzae*, and bacterial amylases and (briefly) phosphorylase. The more than 300 references cited were selected to emphasize the varied character and

modes of action of the amylases. The companion chapter by Kneen and Sandstedt critically discusses amylases in milling and baking technology. The enzymologist as well as the technologist will find this chapter interesting.

The chapter on esterases by Longenecker concerns chiefly lipolytic esterases. Unexpected in a review of this type but of interest to many is a valuable discussion of methods of measuring esterase activity. The twin chapter by Sullivan relative to esterases (lipases and phosphatases) in milling and baking makes a plea for more fundamental experimental data. The phytase-phytin-calcium system is interestingly reviewed.

An excellent pedagogical discussion of classes or types of oxidizing enzyme systems is given by Barron. The companion chapter by Sullivan concerns the occurrence of oxidizing enzymes in wheat rather than their relation to wheat technology, doubtless because of the dearth of definite information concerning such relation. However, in discussing the oxidation-reduction systems in flour and dough and in discussing lipoxidase the possible relation of enzymes to aging and improving flours is considered.

The stimulating review of proteases by Balls and Kies emphasizes the over-all enzymic characteristics as well as the specificity of the proteases. Proteinases and peptidases of plant, animal, and microbial origin receive attention and serve to illustrate the basic principles encountered in protease research. The companion chapter by Hildebrand concerns the role of proteases in baking. A clear presentation of the controversy concerning the mechanism by which oxidizing agents exert an "improving" action in bread baking is given. It is concluded that the bulk of evidence favors a direct action of oxidizing agents on flour proteins rather than an indirect effect involving enzyme inactivation (or, inversely, activation). Indeed, the possibility that proteases play a part in baking, even including proteases of malted wheat, yeast, and insects, has dwindled to such a minor or at best inconclusive stature that, without meaning any criticism, this chapter might have been entitled, "The Lack of Role of Proteases in Baking." On the other hand, it is unlikely that the controversy is dead.

The last two chapters concern fermentation: "The Mechanism of Alcoholic Fermentation" by Werkman and "Yeast Fermentation" by Atkin, Schultz, and Frey. Knowledge of the 10 to 13 main steps in alcoholic fermentation, which are described clearly by Werkman, prepares the reader in part, at least, for the large measure of control procedures necessary in panary yeast fermentation. Thus, temperature, pH, acidity change during yeast fermentation, sugar concentration and type, phosphate and sulfate, magnesium and potassium, nitrogen type, vitamins, and toxic substances are considered. This chapter and perhaps the chapter on amylases in milling and baking technology will be of greatest interest to technologists.

The contents of the book are derived in the main from literature available before 1944—this reviewer noted only an occasional 1944 and no 1945 references. Fortunately, the basic nature of the information presented minimizes this failing. Misstatements, misprints, and omissions are relatively few and do not significantly lower the quality of the work. The book is a valuable one on basic enzymology as well as a valuable source of information on the role of enzymes in wheat technology. Useful author and subject indexes are included. References are given by title as well as by author.

HANS LINEWEAVER
Berkeley, California

Currents in Biochemical Research. Edited by D. E. Green. 500 pp. Interscience Publishers, New York, N. Y. 1946. Price $5.00.

The purpose of this book, as aptly stated in the preface, is "an attempt by some thirty research workers to describe in as simple a language as possible the important developments in their own fields and to speculate a little on the most likely paths of future progress." The success of most of the contributors in attaining this objective makes the book both interesting and stimulating and a useful contribution. It is difficult to give in clear and simple language an accurate evaluation of advances in a technical field. Consequently, several of the essays resemble review articles intended for the specialist. The essays cover far too wide a field for the reviewer to attempt an appraisal of the adequacy of their content. However, the reputation of the contributors should insure the authoritative nature of the presentations.

The contents are as follows: 1. G. W. Beadle, The Gene and Biochemistry. 2. W. M. Stanley, Viruses. 3. H. Gaffron, Photosynthesis and the Production of

Organic Matter on Earth. 4. René J. Dubos, The Bacterial Cell. 5. D. R. Hoagland, The Nutrition and Biochemistry of Plants. 6. C. A. Elvehjem, Biological Significance of Vitamins. 7. Karl Folkers, Some Aspects of Vitamin Research. 8. Donald D. Van Slyke, Quantitative Analysis in Biochemistry. 9. Joseph S. Fruton, Enzymic Hydrolysis and Synthesis of Peptide Bonds. 10. Fritz Lipmann, Metabolic Process Patterns. 11. David E. Green, Biochemistry from the Standpoint of Enzymes. 12. Severo Ochoa, Enzymic Mechanisms of Carbon Dioxide Assimilation. 13. B. A. Houssay, Hormones. 14. Leonor Michaelis, Fundamentals of Oxidation and Reduction. 15. Herman M. Kalckar, Mesomeric Concepts in the Biological Sciences. 16. Max A. Lauffer, Viscometry in Biochemical Investigations. 17. D. Rittenberg and David Shemin, Isotope Technique in the Study of Intermediary Metabolism. 18. Karl Meyer, Mucolytic Enzymes. 19. Konrad Bloch, Some Aspects of Intermediary Metabolism. 20. Gregory Pincus, The Steroid Hormones. 21. Kenneth V. Thimann, Plant Hormones and the Analysis of Growth. 22. David Nachmansohn, Chemical Mechanism of Nervous Action. 23. D. W. Woolley, Some Aspects of Biochemical Antagonism. 24. Rollin D. Hotchkiss, Chemotherapy: Applied Cytochemistry. 25. Arnold D. Welch and Ernest Bueding, Biochemical Aspects of Pharmacology. 26. Charles L. Hoagland, Some Biochemical Problems Posed by a Disease of Muscle. 27. C. H. Best, Physiology and Biochemistry. 28. I. Fankuchen and H. Mark, X-Ray Diffraction and the Study of Fibrous Proteins. 29. Michael Heidelberger, Immunochemistry. 30. W. H. Sebrell, Social Aspects of Nutrition. 31. L. C. Dunn, Organization and Support of Science in the United States.

Some variations in the editorial composition are apparent. They range in length from the tart, eight-page contribution by Heidelberger on immunochemistry to the broader twenty-four page discussion by Gaffron on photosynthesis. Some of the articles have only a few selected references while others have much longer bibliographies.

The reviewer was impressed by the manner in which some potentially complex subjects were presented, as in the articles on oxidation and reduction mechanisms and on X-ray studies of protein structure. Many of the essays, such as those of Dubos, Hoagland, Woolley, and Beadle, provoke interesting speculation as to future developments. The essays by Rittenberg and Shemin and by Bloch on intermediary metabolism are both well written but overlap in many respects.

The emphasis of the book is on the increasing store of fundamental biochemical knowledge, although various writers have clearly stated possible application of newer developments. In particular, Sebrell makes a strong plea for more application of nutritional knowledge.

The book is commendably free from typographical errors; only a few were found in a careful reading. The print and binding are satisfactory. The book merits and undoubtedly will have a good reception.

PAUL D. BOYER
Division of Agricultural Biochemistry
University of Minnesota
University Farm, St. Paul, Minnesota

The Chemistry of the Carbon Compounds. Volume III. The Aromatic Compounds. By Victor von Richter. Edited by Richard Anschütz. Translated from Volume II, part 2 of the 12th German edition (compiled by F. Arndt, A. Butenandt, F. Rochussen, R. Tschesche, A. Weissberger) by A. J. Mee. Elsevier Publishing Co., Inc., New York, N. Y. xviii + 794 pp. Price $15.00.

This translation of the well-known text by Victor von Richter is a welcome and worth-while addition to our shelf of reference books on organic chemistry. *The Chemistry of the Carbon Compounds* has appeared in numerous German and English editions and has been a standard reference for many years. The present volume gives an exhaustive treatment of the field of aromatic compounds and maintains the high standards of previous volumes. Theoretical discussions are limited and not extensive and most of the material deals with the general preparations and properties of aromatic compounds.

The book under review is a translation of the second part of the second volume of the twelfth German edition with the exception of the section on organic free radicals which has been shifted to volume IV. Unfortunately, the war has prevented the

translators from entirely revising and bringing this book up to date as in the case of previous volumes. The first 50 pages have been revised by Dr. T. W. J. Taylor, but the remainder is a literal translation of the original German text without revisions or additions. Thus the literature is complete to 1936.

The book is divided into two main sections: Part I, Mononuclear Aromatic Compounds, Benzene Derivatives (492 pp.); and Part II, Multinuclear Aromatic Compounds (220 pp.). In the first part there are chapters on the mononuclear benzene hydrocarbons, halogen derivatives, nitrogen derivatives, aromatic compounds of phosphorus, arsenic, antimony, bismuth, boron, and silicon, phenyl metallic derivatives, sulfonic acids, phenols, quinones, phenyl paraffin alcohols and their oxidation products, and mononuclear aromatic compounds with unsaturated side chains. Part II is divided into three major divisions dealing with the phenyl-benzene group, polyphenyl paraffins, and condensed nuclei. There is an excellent subject index of 79 pages but no author index.

The arrangement of the book is good and the use of fine type in the descriptive sections permits the inclusion of more material than is indicated by the 794 pages. It is unfortunate that this treatise could not be brought up to date; nevertheless this volume, together with the others of the series, comprises one of the finest references available in the field of organic chemistry.

SIDNEY E. MILLER
General Mills, Inc.
Research Department
Minneapolis, Minnesota

INDEX TO VOLUME XXIII

AUTHORS

SUBJECTS

CONTROL

UNBIASED laboratory tests over a period of several years have conclusively proved that NATIONAL GRAIN YEAST is far superior in the matter of dough control.

And it is because of this and other outstanding qualities that thousands of progressive bakers have come to regard NATIONAL GRAIN YEAST as a primary essential in the art of baking better bread.

NATIONAL GRAIN YEAST CORPORATION

Chanin Bldg., N. Y. C. • Chicago, Ill. • Crystal Lake, Ill. • Belleville, N. J

Frank J. Hale
President

PURE VITAMINS
—Products of Merck Research

Thiamine Hydrochloride U.S.P.
(Vitamin B_1 Hydrochloride)

Riboflavin U.S.P.
(Vitamin B_2)

Niacin
(Nicotinic Acid U.S.P.)

Niacinamide
(Nicotinamide U.S.P.)

Pyridoxine Hydrochloride
(Vitamin B_6 Hydrochloride)

Calcium Pantothenate
Dextrorotatory

Ascorbic Acid U.S.P.
(Vitamin C)

Vitamin K_1
(2-Methyl-3-Phytyl-1,4-Naphthoquinone)

Menadione U.S.P.
(2-Methyl-1,4-Naphthoquinone)
(Vitamin K Active)

Alpha-Tocopherol
(Vitamin E)

Alpha-Tocopherol Acetate

Biotin

Merck & Co., Inc. now manufactures all the vitamins commercially available in pure form, with the exception of vitamins A and D.

Merck research has been directly responsible for many important contributions to the synthesis, development, and large-scale production of individual vitamin factors in pure form.

In a number of instances, the pure vitamins may be considered to be products of Merck research. Several were originally synthesized in The Merck Research Laboratories, and others have been synthesized by Merck chemists and collaborators in associated laboratories.

Because most of the known vitamins have now been made available in pure form, effective therapy of specific vitamin deficiencies can be conducted on a rational and controlled basis, under the direction of the physician.

MERCK VITAMINS

MERCK & CO., Inc. RAHWAY, NEW JERSEY

Manufacturing Chemists

MERCK MERCK

THE ANSWER TO THE LEAVENING PROBLEM

is different

Experience reveals that a leavening which proves most efficient for gingerbread may not be at all satisfactory for a waffle, muffin, cookie, or other type of mix. Furthermore, should the ingredients in a particular cake mix, for example, be varied, then a compensating adjustment must be made in the leavening.

In other words, for best results, *the leavening must be tailor-made for the specific mix in question.*

Today, better than 95% of all chemical leavening includes a phosphate or phosphates combined in varying proportions. The question is which phosphate or phosphates . . . and how much?

The leavening selected must assure maximum shelf life for the mix, and the optimum in volume, lightness, grain, and color of the oven product.

Rate of Reaction Important

Excessive loss of leavening gas while mixing the batter or dough must be avoided. The phosphate or phosphates selected should provide sustained action throughout the oven period without "tailing off" before the end of the bake.

How much leavening also presents a question deserving of careful consideration. Obviously, sufficient leavening should be included to provide maximum expansion in the oven, with a margin of safety to assure adequate shelf life. At the same time an excess must be avoided or the cake is likely to blow-up and fall.

Let Victor Help You

For over forty years Victor Chemical Works has specialized in the production of food phosphates. From Victor's Research Laboratories have come many of the outstanding developments in the science of chemical leavening.

If you would like help in solving the all-important leavening problem, our research laboratory will gladly submit suggestions for the most efficient leavening.

VICTOR PHOSPHATES

For Leavening

Monocalcium Phosphate
"Regent"—(hydrated)
"V-90"—(anhydrous)
Sodium Acid Pyrophosphate
"Victor Cream"
"Perfection"

For Mineralization

Monocalcium Phosphate
Dicalcium Phosphate
Tricalcium Phosphate
Calcium Pyrophosphate
Sodium Iron Pyrophosphate
Iron Orthophosphate
Iron Pyrophosphate

• • •

Experimental samples on request

VICTOR CHEMICAL WORKS

WORLD'S LARGEST MANUFACTURERS OF FOOD PHOSPHATES

141 West Jackson Boulevard Chicago 4, Illinois

GET FAST, CONTROLLED DRYING

for Flax and Soy Tests

with

DESPATCH BUILT

LABORATORY OVENS

***WRITE TODAY** for this helpful new Bulletin 105-CC describing Despatch Laboratory Ovens. Fully illustrated; printed in 2 colors.*

There's no faster, safer, more accurate method of drying flax or soy samples than to run them through a forced-draft *Despatch* Laboratory Oven! Every test gets optimum heat application, accurately controlled.

You can run *oil content determinations* (drying before grinding and re-drying to eliminate solvent), *moisture content tests, constant weight analyses* and scores of other tests with equal ease and accuracy.

FEATURES: Fast heat recovery, 2–8 min. after loading cold samples • Thermostat sensitive to ±1°C. • Wide-range flexibility, 35° to 260°C. • 5 year guarantee of heat elements • Convenient sizes, 13" x 13" x 13" to 37" x 25" x 37"; electric or gas models.

Ask your Dealer for Details

or write direct

DESPATCH OVEN COMPANY
MINNEAPOLIS 14
MINNESOTA, U.S.A.

N-A's nationwide flour treatment service means — prompt shipment from strategically located N-A warehouses, quick response to service calls by N-A Servicemen, assistance on maturing, bleaching and enriching problems and uniform flour treatment results with —

AGENE **NOVADELOX** **N-RICHMENT-A**

WALLACE & TIERNAN CO., INC., Agents for:

NOVADEL-AGENE

N-A 131 BELLEVILLE, NEW JERSEY